MEMORANDUM

INTERMOUNTAIN POWER SERVICE CORPORATION

TO: Joe Hamblin

FROM: Dennis Killian

DATE: April 2, 1997

SUBJECT: Replacement of Rubber-lined Scrubber Slurry Spray Piping

We recommend that we immediately start a replacement program for the rubber lining in the scrubber slurry spray piping. We have already had two failures in the piping system and with the bubbling of the rubber lining, more failures are sure to follow.

We are anticipating that this work will take up to three years to complete if additional manpower is not added to the existing scrubber maintenance crews. This may or may not be soon enough to prevent module availability reductions. It is difficult to predict exactly when the liner will fail because the failure mechanism is not abrasion or erosion. For this reason we also recommend that contingency plans be made to expedite liner repair in the event failures rapidly accelerate.

We also recommend that the liner material specification be changed to ¼-inch black natural rubber instead of the tan natural rubber that was originally installed. This is the least expensive option and has given us good service. The black natural rubber used on the lateral headers and laterals to date does not have the bubbling that is occuring in the tan natural rubber.

Cost
The cost per module to complete the rubber lining in the piping for all three spray pumps will run approximately $40,500. This cost is estimated using $13.00 per ft^2 to apply 2,180 ft^2 of rubber lining and $6.00 per ft^2 to remove the old rubber lining. This does not include the labor required to remove and reinstall the piping in the scrubber. A urethane lining would cost at least double and some bubbling type failures have occured in urethane linings.

Schedule
The current maintenance schedule has been to overhaul a scrubber module about every two months or go completely through all 12 modules in a two year period. With the additional work required

in taking down and reinstalling the slurry piping as well as installing the strainers, installing new laterals when needed and more mist eliminators being replaced a more likely schedule might be a module every three months. This schedule would result in all new rubber-lined pipe within a three year period.

Unless we hear otherwise, we will begin to coordinate with your staff completion of the following action items:

Action Item	Assigned Department
1. Prepare a specification for cleaning and rubber lining.	TS
2. Send out a request for bid for cleaning and rubber lining the piping of one set of piping for one Scrubber Module. Get an alternative bid for urethane coating.	Maintenance
3. Get an estimate and schedule from IPC for removing and reinstalling the piping in the event the schedule needs to be accelerated.	TS
4. Test our current high pressure washers to see how effective they are at removing the old rubber lining. If feasible, determine the economics of purchasing our own very high pressure washer to remove the old rubber lining.	TS & Maintenance
5. Complete an economic analysis of the benefits of purchasing a spare set of scrubber recycle piping to improve relining turn around.	TS
6. Remove the piping from Unit 2 and send to contractor for relining.	Maintenance
7. Complete final plans and schedule for remaining modules	Maintenance

Attached to this memo is a miscellaneous fact sheet detailing some information about the pipe failures and three tables used to calculate the lengths and surface areas of the piping and also three drawings of the piping detailing each run of pipe.

If there are any questions or concerns please contact Jeff Payne at Extension 6439.

JLP:MGN:dh
Attachments

CC: Gale Chapman

Pump 1A is the High Pressure Spray Pump				
Piping Section & Length & Width	Description	Flanges	Connections	Total Square Feet for Rubber Lining
1A-S1 12'4" X 4'8"	From Reaction Tank to Pump Approx. Length 11.8 feet	2	1- Drain reduced to 3"	110.6
1A-S2 7'8" X 3'9"	From strainer to vertical pipe. Approx. Length 7.1 feet	2		74.6
1A-S3 14'6½" X 7' 6½"	Short Vertical with Elbow Approx. Length 14.0 feet Same as 2A-S2 & 3A-s2	2		127.4
1A-S4 6'0½" X 28'6½"	Long Vertical Section Approx. Length 26.5 feet	2		223.1
1A-S5 6'0½" X 12'6½"	Top Horizontal Section with Elbow Approx. Length 10.5 feet	2		100.6
1A-S6 6'0½" X 7'7½"	Horizontal Elbow Connection to Absorber Approx. Length 5.6 feet Same as 2A-S7 & 3A-S7	2		63.1
Totals	Approx. 75.5 ft	12		
Total Area Requiring Rubber Lining				699.4

Pump 2A is the Intermediate Pressure Spray Pump				
Piping Section & Length & Width	Description	Flanges	Connections	Total Square Feet for Rubber Lining
2A-S1 11'0" X 3'7"	From Reaction Tank to Pump Straight Pipe Length 11.0 feet	2	1- Drain reduced to 3"	104.50
2A-S2 7'8" X 3'9"	From strainer to vertical pipe. Approx. Length 7.1 feet Same as 1A-S2 & 3A-S2	2		74.60
2A-S3 14'6½" X 9'9½"	Short Vertical with Elbow Approx. Length 16.25 feet	2		144.70
2A-S4 16'6½" X 8'6½"	Long Horizontal Section with Elbow Approx. Length 17.0 feet	2		150.40
2A-S5 16'0" X 3'7"	Long Vertical Section Straight Pipe Length 16.0 feet	2		142.70
2A-S6 6'0½" X 8'6½"	Short Vertical Elbow Approx. Length 6.5 feet	2		70.00
2A-S7 6'0½" X 7'7½"	Horizontal Elbow Connection to Absorber Approx. Length 5.6 feet Same as 1A-S6 & 3A-S7	2		63.10
Totals	Approx. 79.45 ft	14		
Total Area Requiring Rubber Lining				750.00

Pump 3A is the Low Pressure Spray Pump				
Piping Section & Length & Width	Description	Flanges	Connections	Total Square Feet for Rubber Lining
3A-S1 12'4" X 4'8"	From Reaction Tank to Pump Approx. Length 11.8 feet	2	1- Drain reduced to 3"	110.6
3A-S2 7'8" X 3'9"	From strainer to vertical pipe. Approx. Length 7.1 feet Same as 1A-S2 & 2A-S2	2		74.60
3A-S3 14'6½" X 9'9½"	Short Vertical with Elbow Approx. Length 16.25 feet Same as 2A-S3	2		144.70
3A-S4 18'0½" X 8'6½"	Long Horizontal Section with Elbow Approx. Length 18.6 feet	2		161.90
3A-S5 11'0" X 3'7"	Long Vertical Section Straight Pipe Length 11.0 feet	2		104.50
3A-S6 6'0½" X 8'6½"	Short Vertical Elbow Approx. Length 6.5 feet Same as 2A-S6	2		70.00
3A-S7 6'0½" X 7'7½"	Horizontal Elbow Connection to Absorber Approx. Length 5.6 ft, Same as 1A-S6 & 2A-S7	2		63.10
Totals	Approx. 76.85 ft	14		
Total Area Requiring Rubber Lining				729.40

RESULTS OF SCRUBBER CHEMISTRY CHARACTERIZATION AT THE INTERMOUNTAIN POWER PROJECT (IPP)

Prepared for:

David R. Owens, P.E.
Electric Power Research Institute
3412 Hillview Avenue
Palo Alto, CA 94303

and

James H. Wilhelm
Codan Associates
2394 Charros Road
Sandy, Utah 84092

Prepared by:

Radian Corporation
8501 MoPac Boulevard
Austin, Texas 78759

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Section 1

INTRODUCTION

IPP personnel have experienced scrubber operating problems that appear to be related to process chemistry. These problems include periodic scaling of reaction tank and spray header internals as well as unpredictable changes in solids dewatering properties. Because of the scaling problem, scrubber modules must be shut down and cleaned on a rotating basis. Spray nozzle plugging with chips of hard scale has been particularly troublesome.

As part of the Electric Power Research Institute's (EPRI) FGD Chemical Process Problems Program (Research Project 2248-1), Codan Associates and Radian Corporation participated in a brief study of the IPP wet limestone scrubber operation during November 1990. Important aspects of system design and operation were discussed during a two-day site visit, and a number of process samples were obtained for subsequent off-site analyses. This report summarizes the results of those analyses and discusses the application of the results to possible improvements in scrubber system operation.

Section 2

SUMMARY AND RECOMMENDATIONS

.Results of chemical analyses and subsequent process calculations yielded the following summary of process operating conditions prevalent during the November site visit:

- On-line pH meters were in excellent agreement with local measurements made during slurry sampling. The operating pH ranged from 5.68 to 6.02 in six normally operated scrubber modules.

- Slurry solids content measurements made by the IPP lab using an approximate method based on slurry density were consistently higher than the gravimetric results. The slurry solids content in normally operated scrubber modules ranged from 7.5% to 10.8%. The corresponding IPP lab results ranged from 10.4% to 13.7%.

- The limestone utilization was good (94% to 98%) in normally operated scrubber modules.

- Oxidation was high (90% to 100%) in seven of the eight scrubber modules sampled. The eighth module was in a start-up mode using excess limestone and showed only 8.5% oxidation. Oxidation appears to be inhibited by operation at a pH greater than 6.0.

- Liquid-phase alkalinity in the scrubber slurry greatly exceeded the quantity of SO_2 absorbed.

- The gypsum relative saturation in the recirculating slurry was relatively low (1.14 maximum) in all of the scrubber modules.

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Based on the results of chemical analyses, process calculations, and a brief review of previous IPP laboratory data, the following steps are recommended to improve process operation:

- The difference between slurry solids content results from the gravimetric and slurry density methods should be resolved. Periodic gravimetric determinations should be made concurrently with slurry density measurements until confidence in the slurry density method is re-established.

- The slurry solids content setpoint should be increased from 10% to 15% to increase the gypsum seed crystal quantity and to lessen the impact of operating excursions on slurry solids content. Excess limestone should not be added as a means of increasing the slurry solids content. Excess limestone replaces gypsum seed crystals and thereby increases gypsum scaling potential. It may also adversely affect waste solids dewatering properties by increasing the properties of fines and decreasing the sulfite oxidation rate.

- The scrubber module start-up procedure should be changed so that gypsum seed crystals are introduced to the reaction tank instead of a large excess of limestone. This could be done, for example, by pumping slurry from the waste slurry sump into the start-up module reaction tank instead of the thickener feed tank. The limestone content of the start-up slurry should be reduced to about 0.5% to maintain the start-up pH below 6.0. Any recovered water used to fill the start-up module reaction tank should be mixed with about 20% cooling tower blowdown to lower the gypsum relative saturation.

- The normal operating pH of the scrubber modules should be maintained below 6.0 under all operating conditions. Operation at a pH above 6.0 will tend to lower oxidation and decrease limestone utilization. This will deplete gypsum seed crystals in the reaction tank, increase the likelihood of scaling events, and change the solids dewatering characteristics. Since the on-line pH meters appear to be working well, consideration should be given to operating in a straight pH control mode without feed-forward bias. Whenever the operating pH must be controlled above 6.0 to achieve the desired SO_2 removal efficiency, it is recommended that the faulty module be taken off line to remove nozzle obstructions and scale. Doing so will

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help avoid poor limestone utilization and potential scale formation.

- Once gypsum scale deposits have formed in lines and on vessel walls, the scale will continue to grow even at the low gypsum relative saturations measured. If the measures recommended above do not satisfactorily reduce the pluggage of nozzles and other operating and maintenance problems associated with scale formation, it is recommended that the vessel walls and lines be cleaned to completely remove all scale. It is expected that, if the gypsum relative saturation is maintained below about 1.3 by using the measures listed above, new scale deposits will not form.

- It is recommended that periodic tests be performed every three to six months to evaluate scrubber chemistry and control. Results should be compared to those in this study, especially for gypsum relative saturation, scrubber pH, scrubber suspended solids concentration, limestone utilization, and percent oxidation.

- It is expected that the periodic slugging of solids contact unit (SCU) slurry into the scrubbing system could have a detrimental effect on limestone utilization due to possible spikes in the magnesium concentration. Therefore, it is recommended that the blowdown of SCU slurry be continuous and uniform.

Section 3

SUMMARY SAMPLING AND ANALYTICAL RESULTS

Table 3-1 is a summary of the process samples obtained during the site visit in November 1990. A complete set of slurry samples for chemical analyses was obtained from each of the four individual scrubber modules of both Units 1 and 2 during the afternoon of 11/19/90 when the units were operating at high load (about 800 MW). Unit 2 was sampled again early the following morning after about 5 hours of operation at low load (400 MW). Slurry samples for weight percent solids analyses were also obtained from Unit 1 scrubber modules early on 11/20/90. A supplemental sample from module 2D was obtained on 12/11/90 by IPP and shipped to Radian.

In addition to the samples from the recycle slurry of the individual scrubber modules, samples of cooling tower blowdown and recovered water which provide makeup to the scrubber systems were obtained from hose bibs located inside the scrubber buildings. The reagent slurry and solids contact unit (SCU) under-flow slurry were sampled from storage tank outlets inside the reagent preparation building.

The slurry sampling technique and analytical methods were similar to those described in EPRI CS-3612, FGD Chemistry and Analytical Methods Handbook. A syringe was used to draw slurry from the pH control pots of the scrubber modules. The slurry samples were immediately filtered, and the filtrate was introduced to four individual tared bottles containing deionized water and reagents specific to the liquid-phase analytical methods. The collected fil-trates were diluted in this sampling process so that precipitation would not

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Table 3-1

SCRUBBER PROCESS SAMPLE SUMMARY

Unit	Date	Load	Sample Location	Sample Time
1	11/19/90	830 MW	A Recycle	1312
			B Recycle	1330
			E Recycle	1350
			F Recycle	1403, 1412
			Recovered Water	1340
			Reagent Tank	1440
			Cooling Tower	
			Blowdown	1430
			SCU Sludge	1450
2	11/19/90	790 MW	A Recycle	1550
			B Recycle	1600
			D Recycle	1610
			E Recycle	1615
			Cooling Tower	
			Blowdown	1540
1	11/20/90	830 MW	A Recycle*	0610
			B Recycle*	0615
			D Recycle*	0620
			E Recycle*	0625
2	11/19/90	400 MW	B Recycle	0530
			D Recycle	0545
			E Recycle	0555
2	12/11/90	800 MW	D Recycle	0900**

*Sampled for wt.% solids only.

**Sample taken by IPP.

occur prior to the analyses. A fifth tared bottle was filled with unfiltered slurry for determination of slurry solids content and subsequent chemical analyses of the solids portion of the slurry. The pH and temperature of each slurry sample were also measured using a portable pH meter.

Results of chemical analyses of slurry samples from Units 1 and 2 are summarized in Tables 3-2 and 3-3. As a check on the overall quality of the analytical data, molar charge imbalances were calculated for each data set. The charge imbalance is expressed as a percentage representing the sum of the products of the individual species concentrations and their charges divided by the total of the species concentrations. If all of the analyses are accurate and no chemical species are unaccounted for, then the charge imbalance should be zero. A charge imbalance of ±5% or less is considered acceptable.

In general, the calculated charge imbalances indicate good sampling and analytical data quality with the exception of a few of the liquid-phase data sets. Results from liquid samples 1A, 1F, and 2D (low load) clearly exceed the acceptable charge imbalance criterion. There does not appear to be a systematic positive or negative error in the liquid charge imbalances, indicating that all significant species have been included in the analyses. The most likely source of error in the results is contamination of the filtrate samples with slurry solids during the sampling procedure. Since the cations and anions are determined using separate sample bottles, introduction of solids to either bottle will result in a charge imbalance for the data set even though the solids themselves are neutral.

As an additional check on the reproducibility of the sampling and analytical procedures, two sequential sample sets were obtained from module 1F. Results for these duplicate samples were in good agreement except for the chloride analyses. Since chloride cannot be affected by solids contamination, this difference must be due to some other analytical error. Since the second of

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Table 3-2

RESULTS OF CHEMICAL ANALYSES - UNIT 1

Sample	1A Recycle	1B Recycle	1E Recycle	1F Recycle	1F Recycle	Recovered Water	Reagent Feed	CTB*	SCU**
Date	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90
Time	1312	1330	1350	1403	1412	1340	1440	1430	1450
Measured pH	5.86	6.39	5.89	5.68	5.77	6.96	9.29	7.07	10.14
Indicated pH	5.89	6.39	5.81	5.70	5.75	N.A.	N.A.	N.A.	N.A.
Temperature (°C)	48.2	47.8	47.5	47.8	47.8	15.0	27.8	N.A.	14.1
Liquid-Phase Analyses (mg/L)									
Ca	648	860	621	641	637	645	352	442	92
Mg	1440	1510	2180	2130	2120	1670	407	257	100
Na	4010	3820	5940	6140	5750	5110	2360	1210	710
Cl	5380	4650	9030	5390	8160	7570	2720	1740	925
CO_3	186	241	254	199	218	151	N.A.	N.A.	N.A.
SO_3	723	969	865	446	431	24	N.A.	N.A.	N A.
SO_4	9890	7110	11800	11000	11000	10100	3310	2080	913
Charge Imbalance (%)	-8.1	+6.2	-6.0	+12	-1.3	-4.9	+2.9	+2.2	-1 9
Solid-Phase Analyses (mg/g)									
Ca	235	362	239	236	224	N.A.	379	N.A.	314
Mg	1	6	2	2	1	N.A.	13	N.A.	58
CO_3	13	450	20	10	8	N.A.	574	N.A.	472
SO_3	0	98	46	0	0	N.A.	0	N.A.	0
SO_4	538	11	478	547	548	N.A.	2	N.A.	5
Molar Imbalance (%)	+0.9	+2.6	+1.3	+1.0	-1.5	N.A.	+2.3	N.A.	+12.7
Total Suspended Solids (wt.%)	7.52	3.28	9.98	10.21	10.21	N.A.	31.78	N.A.	15.28
Inert Solids (wt.%)	0.48	1.80	0.56	0.76	0.60	N.A.	0.76	N.A.	0.93

*Cooling Tower Blowdown.

**Solids Contact Unit (underflow from raw water treatment).

N.A. = not analyzed.

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Table 3-3

RESULTS OF CHEMICAL ANALYSES - UNIT 2

Sample	2B Recycle	2D Recycle	2B Recycle (low load)	2D Recycle (low load)	2E Recycle (low load)	2D Recycle	Cooling Tower Blowdown
Date	11/19/90	11/19/90	11/20/90	11/20/90	11/20/90	12/11/90	11/19/90
Time	1600	1610	0530	0545	0555	0900	1630
Measured pH	5.80	6.25	5.88	6.34	6.02	6.0	7.00
Indicated pH	5.75	6.22	5.78	6.45	5.87	5.8	N.A.
Temperature (°C)	48.7	48.4	44.5	44.9	44.5	48	
Liquid-Phase Analyses (mg/L)							
Ca	774	681	697	693	637	624	340
Mg	1920	2070	2180	2070	2020	2300	220
Na	5770	5810	5500	6030	6060	7020	1590
Cl	7990	7860	8310	6370	7750	9610	2140
CO_3	217	392	260	334	188	226	N.A.
SO_3	1160	514	181	424	263	1170	N.A.
SO_4	11000	10600	10800	8840	10700	12300	2610
Molar Charge Imbalance (%)	-3.5	+0.1	-1.4	+13	+1.9	-2.7	-5.4
Solid-Phase Analyses (mg/g)							
Ca	234	249	249	233	227	248	N.A.
Mg	2	3	2	2	2	3	N.A.
CO_3	13	82	47	7	9	12	N.A.
SO_3	0	0	0	0	0	147	N.A.
SO_4	539	475	508	541	542	404	N.A.
Molar Imbalance (%)	+0.8	+0.4	+1.9	+1.3	-0.4	+0.5	N.A.
Total Suspended Solids (wt.%)	10.78	13.96	10.46	13.85	9.80	13.41	N.A.
Inert Solids (wt.%)	0.76	1.52	1.72	1.48	0.64	0.82	N.A.

N.A. = not analyzed.

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the two samples has a low charge imbalance, it appears to have the correct chloride concentration.

The solids analyses show excellent charge balance results. The only solids data set exceeding the ±5% criterion is the SCU solids. This sample probably contains magnesium hydroxide, however, and the hydroxide ion has not been included in the analyses. If the observed magnesium is assumed to be exclusively magnesium hydroxide, then the charge imbalance for this data set is less than 1%.

Section 4

COMPARISON OF MEASURED AND INDICATED RECYCLE SLURRY pH VALUES AND SOLIDS CONTENTS

The slurry pH indicated by the on-line pH meter was recorded at each sample point after measuring the slurry pH with a calibrated portable pH meter. The weight percent solids content of the slurry indicated by the on-line density meter was also recorded at each sample point. Table 4-1 compares measured and indicated slurry pH data as well as weight percent solids data determined by analyses at Radian Corporation, routine analyses by the IPP lab, and on-line density meters.

The agreement between measured and indicated pH values at the slurry recycle pH pot sampling points was excellent. The results showed that the on-line pH analyzers were well-operated and maintained and that the current calibration schedule was adequate.

Results for slurry solids content determined from the Radian samples did not agree well with either the on-line density meters or the routine IPP lab results. The density meters did not appear to show a systematic deviation from the Radian results. The 1A density meter agreed well on both sampling occasions. One of the new meters being tested on the 1F module also showed good agreement. About half of the other meters showed higher results and half showed lower results than the Radian samples. For the meters that did not agree well with Radian results, the average deviation was about 35%.

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Table 4-1

COMPARISON OF MEASURED AND INDICATED RECYCLE SLURRY pH VALUES AND SOLIDS CONTENTS

| | | | Slurry pH | | Slurry Solids Content (wt.%) | | IPP Lab | |
Sample	Date	Time	Measured	Indicated	Radian Result	Density Meter	0900	2100
1A	11/19/90	1312	5.86	5.89	7.52	7.5	10.6	10.1
1B	11/19/90	1330	6.39	6.39	3.28	1.0	5.7	5.3
1E	11/19/90	1350	5.89	5.81	9.98	12.5	13.5	14.0
1F	11/19/90	1403	5.68	5.70	10.21	Xertex 12.5*	13.7	12.5
1F	11/19/90	1412	5.77	5.75	10.21	Monitek 10.9*		
2A	11/19/90	1550	5.85	5 88	9.40	6.7	12.0	
2B	11/19/90	1600	5.80	5 75	10.78	14.2	13.7	13 7
2D	11/19/90	1610	6.25	6 22	13.96	10.5	16.1	17 7
2E	11/19/90	1615	5.87	5.81	10.29	13.5	13.6	12 3
1A	11/20/90	0615	N.A.	N A.	7.28	7.1		
1B	11/20/90	0620	N.A.	N.A.	3.34	1.4		
1E	11/20/90	0625	N.A.	N.A.	9.68	12.5		
1F	11/20/90	0630	N.A.	N.A.	9.98			
2B	11/20/90	0530	5.88	5.78	10.46	15**		
2D	11/20/90	0545	6.34	6.45	13.85	10.8		
2E	11/20/90	0555	6.02	5.87	9.80	13.4		
2D	12/11/90	0900	6.0	5.8	13.41			18.4

*Local Indicators - control room indicator not operating.

**Indicator pegged at 15% maximum.

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The IPP laboratory routinely determines the slurry solids content for each scrubber module twice each day. These results are also shown in the table. Even though the IPP lab samples were not obtained at exactly the same time as the Radian samples, the rate of change in the solids content should be small enough that the results can be compared. As can be seen from the data, the IPP lab results were consistently higher than the Radian lab results by 2 or 3 percentage points.

The Radian data are based on drying and weighing the solids from a slurry sample bottle that is weighed before and after filling. Assuming that a representative slurry sample is obtained, this gravimetric method is extremely accurate and reproducible. Results for two sequential samples taken from module 1F, for example, were the same to four significant figures.

The IPP lab results are used on a daily basis to adjust the recovered water makeup rate to the scrubber reaction tanks to control solids density. For this reason, a more rapid turnaround is required than that afforded by the straight gravimetric method. In the method used by IPP, the weight gain of a 100 mL graduated cylinder is measured, and the calculated slurry density is compared to a calibration curve (developed using the more precise gravimetric method) to yield a direct estimate of the slurry solids content. Based on the comparison of results in Table 4-1, it appears that the solid and/or liquid densities have changed since the solids content versus slurry density curve was developed.

Section 5

CALCULATION OF PROCESS PARAMETERS USING SLURRY ANALYSES

Results of liquid-phase analyses were input in the EPRI FGDLIQEQ computer
program to calculate relative saturations of dissolving and precipitating spe-
cies. This program also calculates the alkalinity of the liquor with respect
to SO_2 sorption. Results of the solid-phase analyses were used to calculate
the percentage of absorbed SO_2 that is oxidized and the percentage of lime-
stone utilization. Table 5-1 summarizes the results of calculated process
parameters based on slurry analyses.

LIMESTONE UTILIZATION

Limestone utilization was calculated from the solids analyses by dividing the
sum of the sulfite and sulfate molar contents by the sum of sulfite, sulfate,
and carbonate molar contents. Alternately, limestone utilization is equal to
the difference of calcium and carbonate contents divided by the calcium con-
tent. The results shown in Table 5-1 are an average of these two calculated
values. Since the carbonate analyses may include some dolomite which is not
normally soluble under FGD conditions, utilization based on available carbo-
nate may be slightly higher than indicated.

Limestone utilization appeared to be very high in all of the modules with the
exception of 1B, which was in a start-up mode when sampled, and 2D, to which
excess limestone had been added in an attempt to increase the slurry solids
content for test purposes. Utilization decreased overnight in module 2B from
96% to 87% after operation at reduced load. This decrease was not observed in

Table 5-1

PROCESS PARAMETERS CALCULATED USING SLURRY ANALYSES

Sample	1A	1B	1E	1F	1F (Dup)	2B	2D	2D	2B (low load)	2D (low load)	2E (low load)	Recovered Water	Unit 1 CTB	Unit 2 CTB
Date	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	11/19/90	12/11/90	11/20/90	11/20/90	11/20/90	11/19/90	11/19/90	11/29/90
Time	1312	1330	1350	1403	1412	1600	1610	0900	0530	0545	0555	1340	1430	1540
pH	5.86	6.39	5.89	5.68	5.77	5.80	6.25	6.0	5.88	6.34	6.02	6.96	7.1	7.0
Weight % Solids	7.5	3.3	10.0	10.2	10.2	10.8	14.0	13.4	10.5	13.9	9.8	--	--	--
Relative Saturations														
$CaCO_3$	0.04	0.4	0.05	0.02	0.03	0.04	0.3	0.06	0.06	0.3	0.06	0.2	NA	NA
$CaCO_3 \cdot 1/2H_2O$	3.3	11	3.6	1.5	1.6	5.3	3.8	5.3	0.9	3.4	1.5	0.3	0	0
$CaSO_4 \cdot 2H_2O$	1.04	1.05	0.95	1.01	0.98	1.14	1.00	0.90	1.08	0.90	0.99	1.24	0.6	0.6
Limestone Utilization (mole %)	96	16	94	97	98	96	78	97	87	98	97	NA	NA	NA
Oxidation (mole %)	100	8.5	90	100	100	100	100	70	100	100	100	NA	NA	NA
Liquid-Phase Alkalinity (mmol/L)	4.2	10.2	5.9	3.4	3.4	5.9	8.0	8.1	3.4	7.4	3.6			
(% of inlet SO_2)	380	930	540	310	310	540	730	740	310	670	330			

Module 2E at reduced load. No explanation for this difference is available based on the limited amount of data obtained.

Test results at EPRI's High Sulfur Test Center (HSTC) pilot unit and at other full-scale limestone wet scrubbing systems have shown that, for a given hold tank size, reagent grind, and slurry chemistry, limestone utilization is primarily a function of scrubber recycle slurry operating pH. HSTC data, for example, showed a decrease in limestone utilization from 94% to about 70% as the slurry pH increased from 5.3 to 5.7.

HSTC test data also showed that the dissolved calcium concentration had a strong effect on limestone utilization through its effect on calcium carbonate relative saturation. Higher dissolved calcium concentrations tend to decrease limestone utilization at a given operating pH. The HSTC baseline tests were conducted primarily with a dissolved calcium content of 140 mM. The IPP analytical results showed a dissolved calcium concentration in the range of 15 to 20 mM, which is quite low. The observed low dissolved calcium concentration in the IPP scrubber liquor appears to be a result of the common ion effect of a relatively high dissolved sodium concentration.

The IPP scrubber system has a relatively large reaction tank volume. The estimated average solids residence time for full-load operation is about 100 hours versus 16 hours for the HSTC pilot unit.

The net result of the large hold tank and low dissolved calcium concentration in the IPP scrubber system is to promote high limestone utilization at relatively high operating pH levels. This is illustrated in Figure 5-1 which shows the observed relationship between limestone utilization and operating pH at IPP during the Codan/Radian site visit. This figure shows the expected trend of decreasing utilization with increasing pH, but the permissible range of operating pHs for good utilization is much higher than that seen at the
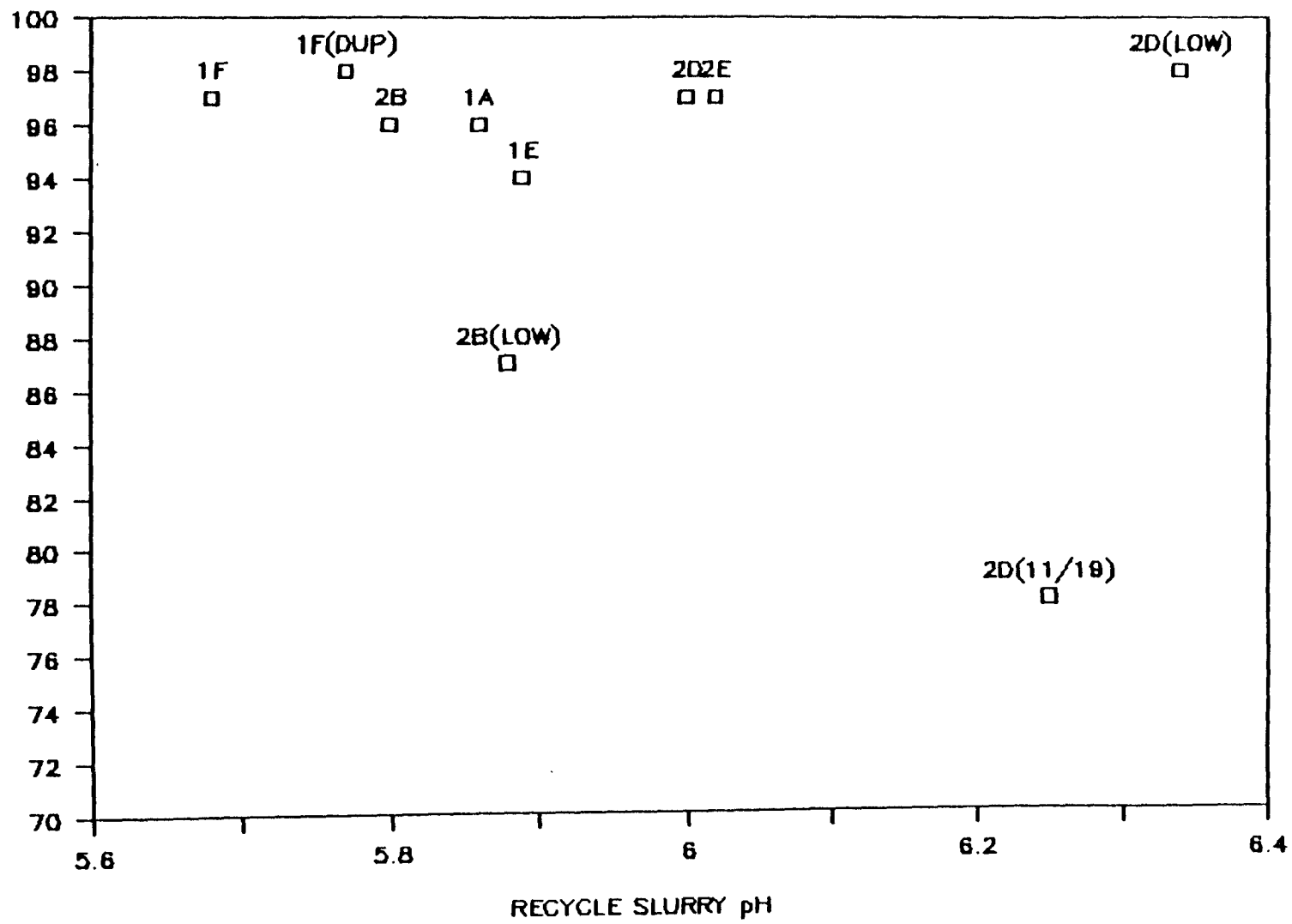
Figure 5-1. Utilization vs. pH

HSTC. It should be noted that the module 2D (low load) data point appears anomalous, but no explanation has been found for this result.

OXIDATION

The percentage of absorbed SO_2 that was oxidized was calculated from the solids analyses by dividing the molar sulfate content of the solids by the sum of the molar sulfite and sulfate contents. Results of these calculations showed that oxidation was 100% in eight of the eleven scrubber recycle slurry samples analyzed. This result is not unexpected for a limestone wet scrubber in a low-sulfur coal application. Oxidation is favored by a high ratio of oxygen to sulfur dioxide in the flue gas.

Two of the 11 slurry samples had significantly lower oxidation percentages. Module 1B was in a start-up mode and had been operating less than one day when the slurry was sampled. The calculated oxidation for this module was only 8.5%. The oxidation reaction in limestone wet scrubbing systems is not completely understood. The presence of trace quantities of dissolved metal species is known to catalyze the reaction. The solubility of these metals decreases with increasing pH. In the case of the start-up module, 1B, the only major difference in the liquid-phase analyses compared to module 1A, for example, is the pH. Based on the results for the start-up module sample, it appears that oxidation is inhibited by operation with excess limestone at a higher-than-normal pH in the IPP scrubber modules.

The next lowest oxidation was 70% for the module 2D slurry that was sampled by IPP on 12/11/90. This module had been operated at a higher-than-normal slurry density as part of the test plan. The limestone utilization for the module 2D sample was high (about 97%), and the operating pH was normal (about 6.0). At first glance, lower oxidation for this sample would not appear to be related to operation with excess limestone. Interpretation of these results is complicated by the long solids residence time in the reaction tanks, however.

The slurry solids content in module 2D had been initially increased by adding an excess of limestone just prior to the 11/19/90 sampling period. At that time, the slurry solids analyses showed 100% oxidation. However, since the average solids residence time in the reaction tank is about 100 hours, the samples obtained from module 2D on 11/19 and 11/20 would represent solids that had been produced prior to operation with excess limestone. Similarly, the sample taken by IPP on 12/11/90 could represent solids that had been produced during operation with excess limestone even though the limestone utilization was high when the sample was taken.

LIQUID-PHASE ALKALINITY

The EPRI FGDLIQEQ computer program was used to calculate the liquid-phase alkalinity of the scrubber recycle slurry samples using data from the liquid-phase analyses as input. The calculated liquid-phase alkalinity in mM is a measure of the ability of the slurry to absorb SO_2 in the absorber without dissolution of additional limestone solids. Major contributors to alkalinity include the sulfite and bicarbonate ions and their soluble complexes with calcium, magnesium, and sodium.

Results of the alkalinity calculations are included in Table 5-1. These results have also been plotted versus pH in Figure 5-2. This figure shows how liquid-phase alkalinity is increased at high pH due to the shift in equilibria from bisulfite to sulfite and from carbonic acid to bicarbonate. Also shown on the figure is an estimate of the required liquid-phase alkalinity based on 100% removal of 400 ppmv of SO_2 in the inlet gas and an L/G of 60 gal/1000 acf in the scrubbers. It is apparent that all of the liquid samples have excess alkalinity. This is another reason why high limestone utilization can be achieved at IPP without any adverse affect on SO_2 removal efficiency.
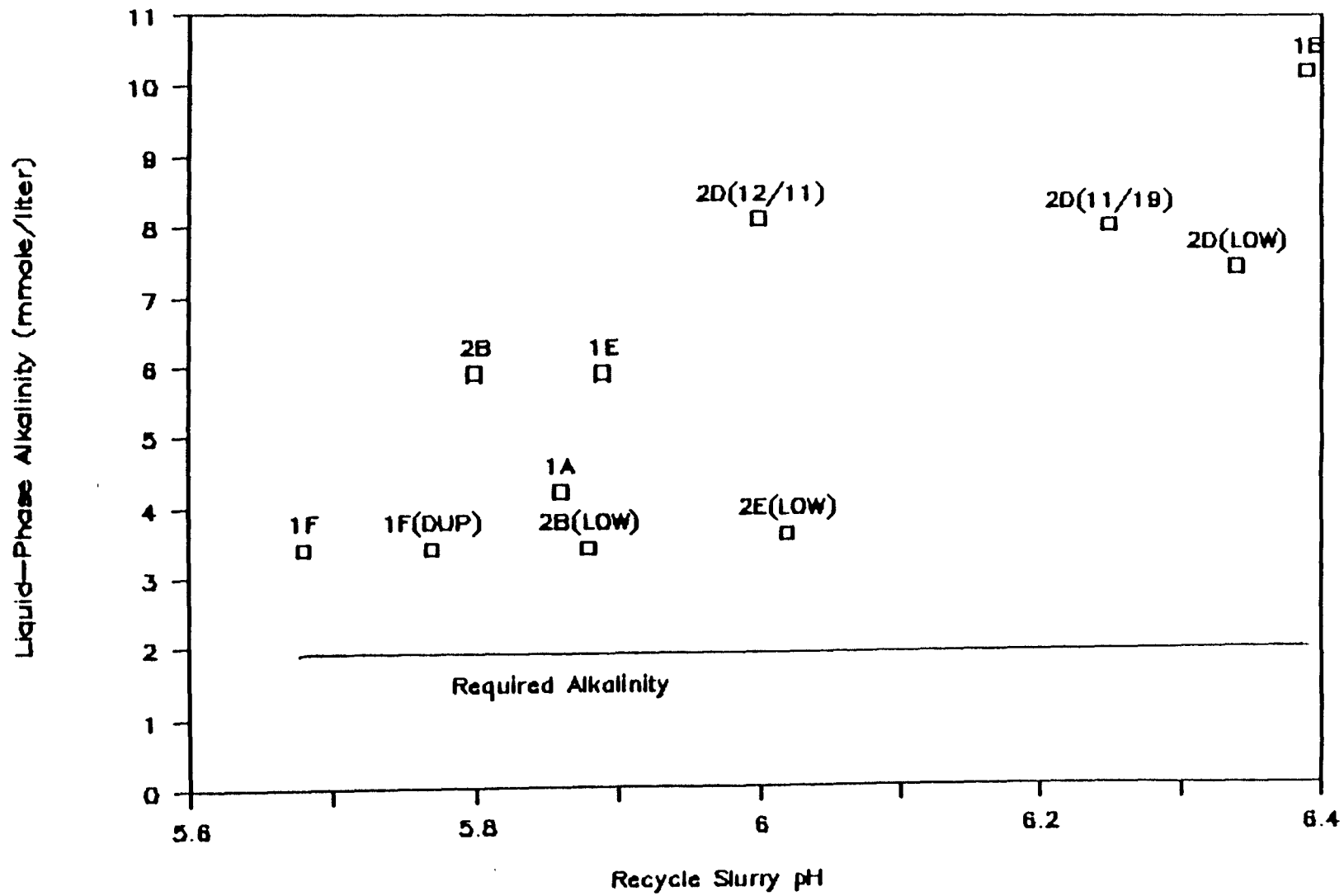
Figure 5-2. Alkalinity vs. pH

RELATIVE SATURATIONS

Relative saturation is defined as the ratio of the liquid-phase activity product of a dissolving or precipitating solid to its equilibrium solubility product. If the relative saturation is greater than 1.0, the solid will tend to precipitate; if it is less than 1.0, dissolution will occur. The EPRI FGDLIQEQ results for calcium carbonate, calcium sulfite, and calcium sulfate dihydrate (gypsum) are summarized in Table 5-1.

EPRI/HSTC data have shown that the dissolution rate of limestone is a strong function of the calcium carbonate relative saturation for a given process configuration. This relationship is also apparent from the results shown in Table 5-1. Only three of the eleven recycle slurry data sets show significantly high calcium carbonate relative saturations. The highest, 0.4 for sample 1B, is in the module with the lowest limestone utilization. The next highest, 0.3 for sample 2D, is in the module with the next lowest limestone utilization. The data for the module 2D low load sample with a relative saturation of 0.3 and a limestone utilization of 98% again appear anomalous.

The highest calcium sulfite relative saturation was 11 for module 1B, which was the start-up module showing very low oxidation. Other modules showed calcium sulfite relative saturations as high as 5.3 without any calcium sulfite solids present in the slurry, but this is not unreasonable since calcium sulfite can attain high relative saturations without nucleating. Considering that the average solids residence time in the reaction tanks is about 100 hours, it is also possible that the conditions prevalent in the liquid phase when the samples were taken were not representative of the average conditions under which the solids were precipitated.

The calcium sulfate relative saturation is of most interest from the standpoint of scaling problems. Sulfate scale is likely to be a problem when the relative saturation of gypsum is greater than 1.3 to 1.4. Since the

permissible operating range of calcium sulfate relative saturation is narrow, some additional analytical effort was made to improve the accuracy of the calculations. A portion of the module 1A liquid sample was equilibrated with gypsum crystals and re-analyzed. The results of these analyses were input to the computer program, and the calcium sulfate relative saturation was calculated. This equilibrium sample had a calculated calcium sulfate relative saturation of 0.92. The calculated results for the remaining samples were adjusted by dividing them by 0.92. This adjustment accounts for minor inaccuracies inherent in the computer program.

Calculated calcium sulfate relative saturations (after adjustment) ranged from 0.90 to 1.14 in the recycle slurry samples. These results indicate that none of the scrubber module reaction tanks should have been in a scaling condition when sampled. The fact that relative saturations less than 1.0 were calculated for modules in which gypsum was obviously precipitating reflects the difficulty of accurate determination of this parameter. The combination of small errors in the seven separate chemical analyses and inaccuracies in the computer program itself can easily account for results that are 10% subsaturated.

Figure 5-3 is a plot of gypsum relative saturation as a function of the suspended solids in the scrubber recycle slurry. While there is some scatter in the data, a linear curve fit of the data suggests that the relative saturation for gypsum is lower at higher concentrations of suspended solids. This trend is consistent with experience at other scrubber installations. The higher surface area provided by the higher concentration of suspended solids tends to promote the precipitation of gypsum on the existing crystals and reduces the driving force for deposition of gypsum on the other surfaces within the scrubbing system.
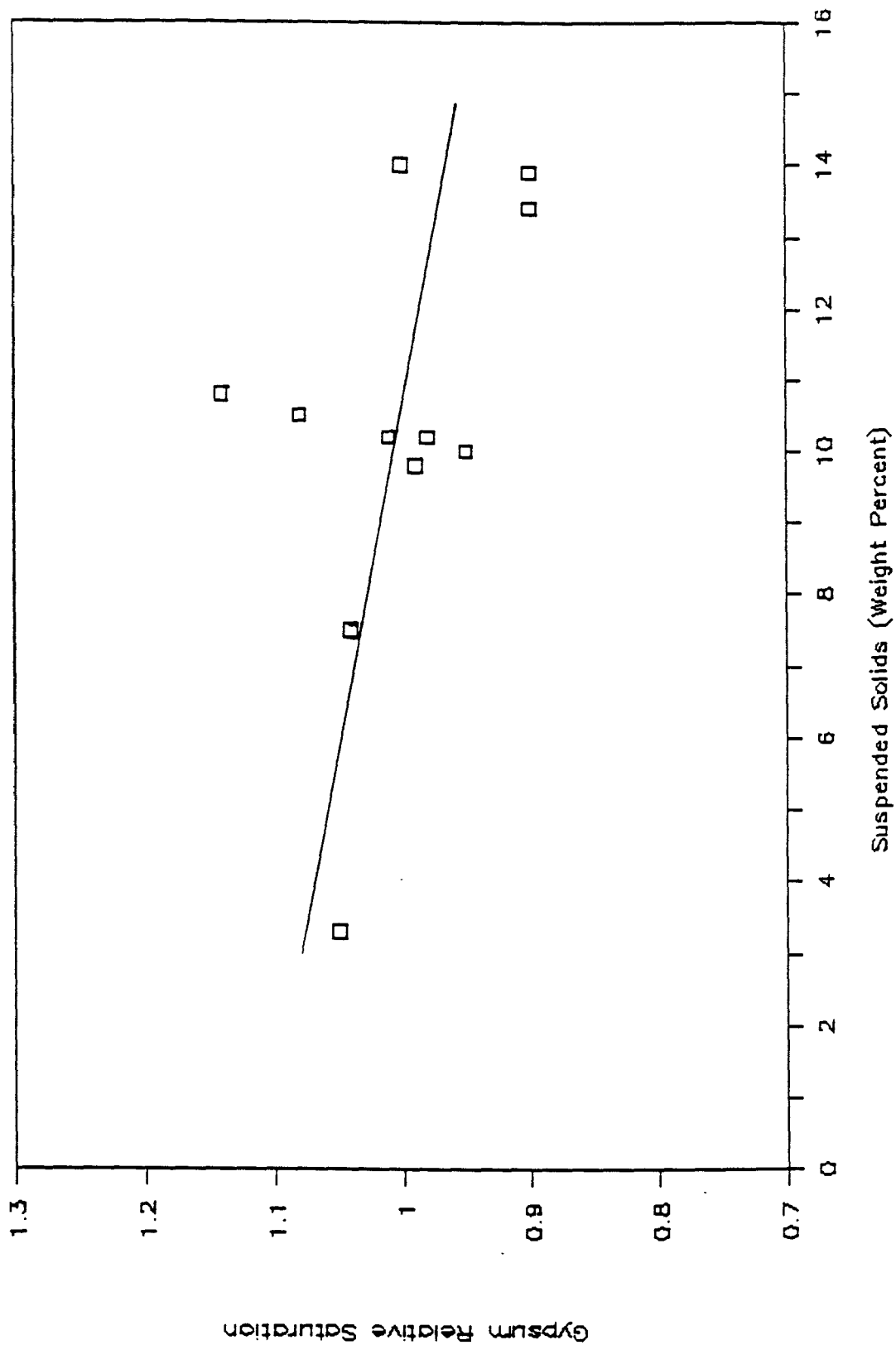
Figure 5-3. Gypsum Relative Saturation vs. Slurry Suspended Solids

IP12_006376

The trend illustrated in Figure 5-3, along with experience at other scrubbing systems, suggests that the concentration of suspended solids in the recycle slurry should be kept at 14 to 16 wt.%. Some systems have operated as high as 20 wt.%, but as the solids concentration increases, there is a tendency for more abrasion, so very high concentrations are not recommended.

The highest calculated gypsum relative saturation of 1.24 was for the recovered water sample. This is consistent with results seen at other limestone scrubbing systems. Additional oxidation of dissolved sulfite to sulfate in the thickener can increase the gypsum relative saturation after the seed crystals have been separated from the slurry.

The lowest calculated gypsum relative saturations were 0.6 for the Units 1 and 2 cooling tower blowdown samples. These results showed that this liquid was of adequate quality for its current use in mist eliminator washing and pump seals.

OVERALL SYSTEM MAGNESIUM BALANCE

In the IPP scrubber system, underflow sludge from the raw water pretreatment thickener (solids contact unit or SCU) is combined with reagent limestone in the grinding circuit. This SCU sludge contains precipitated magnesium hydroxide which presumably dissolves in the scrubber circuit. IPP requested that the potential effect of this sludge on scrubber process chemistry be evaluated. Dissolved magnesium can be beneficial from the standpoint of increasing the liquid-phase alkalinity of the scrubber liquor, but it can also have an adverse affect on limestone utilization. To examine the relative effect of the SCU sludge, the solid and liquid-phase magnesium analyses can be combined with system flow rates to estimate the relative contribution of the SCU sludge to the overall system magnesium balance.

The primary liquid stream entering the scrubber system was cooling tower blow-down (CTB). At high load, an average of about 675 gpm of CTB was flowing into the Unit 1 scrubber system (measured by the mist eliminator wash tank total-izer). The magnesium content of the Unit 1 CTB was 10.5 mM. The product of these terms is 27 moles/min of magnesium entering the Unit 1 scrubber through the mist eliminator wash.

The total reagent slurry flow rate to Unit 1 was not measured, but it can be estimated from the inlet SO_2 quantity derived from a combustion calculation. The limestone molar flow rate should be roughly equal to the inlet SO_2 flow rate which is about 750 g-moles/min or roughly 75,000 g/min of reagent solids. The reagent solids (including SCU sludge) magnesium content was 0.55 mmol/g. The product of these terms is about 41 moles/min of magnesium entering the Unit 1 scrubber system with the reagent feed solids. If the limestone is 5% by weight dolomite, then 10 moles/min of this magnesium is from the limestone and 31 moles/min is from the SCU sludge.

The above calculations show that the magnesium entering the Unit 1 scrubber system from the SCU sludge is roughly equal to that entering from the CTB used to wash the mist eliminator. Some additional magnesium may dissolve from the dolomite component of the limestone, but this is probably a small percentage of the total amount entering from the CTB and SCU sources. Since the recycle slurry solid-phase analyses indicated generally good limestone utilization, the SCU sludge does not appear to have a detrimental effect on the system and may be beneficial with respect to SO_2 removal efficiency.

The total magnesium entering each of the scrubber systems at IPP is about 60 moles/min, not including the dolomite content of the limestone. The amount of magnesium leaving each system in the waste solids is much less than this (about 13 moles/min). This observation may indicate that the steady-state concentration of dissolved magnesium in the system has not yet been reached.

5-12

Section 6

CHARACTERIZATION OF SOLIDS

Various tests were performed to evaluate the characteristics of the solids produced in the scrubbing system. Scanning electron photomicrographs (SEM's) were made by electron microscope, and particle size distributions were determined using a Microtrac Particle Size Analyzer. The SEM's and the output from the Microtrac are included in Appendix A.

The SEM's revealed that there was considerable particle abrasion or attrition as the slurry was recirculated within the scrubbing system. Normally, gypsum particles appear as rhombic columns with sharp edges in SEM's. Most of the gypsum particles in these SEM's have rounded corners, and many appear to have an oval or football shape. The normally sharp edges have probably been rounded off by knocking against other particles and surfaces during the relatively long residence time in the system.

The particles taken from module 1B on 11/19/90 during start-up of this module were mostly very small calcium sulfite crystals mixed with limestone. There also appeared to be some gypsum in this sample even though the solids analyses suggested that only sulfite/sulfate solid solution should be present. The small limestone particles were also evident in the samples from module 2D, which was being operated at a high pH with excess limestone added to increase the concentration of suspended solids. It is evident that these two operating conditions would produce sludge that would not dewater well due to the small size of the particles.

6-1

IP12_006379

The particle size and settling rate data are summarized in Table 6-1, which gives mean particle sizes and the amount of fines as characterized by the percentage of minus 9.4 micron particles, and the initial settling rate of the slurry. The data are consistent with the SEM's discussed above. It is evident that the settling rates were decreased by several orders of magnitude when a large amount of limestone fines was present. The small amount of calcium sulfite particles in sample 1E was enough to increase the amount of fines but had a negligible influence on the settling rate.

Figure 6-1 is a plot of mean particle sizes as a function of suspended solids concentration in the recycle slurry. It is apparent that the particles do not grow much larger than a mean diameter of 75 to 80 microns. Lower suspended solids concentrations, which correspond to shorter residence times for solids in the scrubber system, result in smaller particles--especially on start-up.

Figure 6-2 is a plot of initial settling rates as a function of the percentage of small particles. Again, the settling rates were all excellent except for the three samples that had an excess of limestone.

It was noted that there was a certain amount of darker, fine material in most of the samples. These fines are not a problem in the scrubber slurry with the small percentage present, but if they are allowed to accumulate by recycle in the dewatering system, they are expected to cause dewatering problems.

Table 6-1

SUMMARY OF PARTICLE CHARACTERIZATION DATA

Sample	Mean Particle Size (μ)	% Minus 9.4μ Particles	Initial Settling Rate (ft/min)
1A - 11/19	73.1	4.2	0.33
1B - 11/19	29.5	73	0.006
1E - 11/19	68.5	6.0	0.64
1F - 11/19	77.4	3.4	0.64
2A - 11/19	75.0	3.4	0.21
2B - 11/19	80.0	2.3	0.64
2D - 11/19	78.0	11.4	0.003
2E - 11/19	75.6	3.1	0.32
2B - 11/20	76.8	2.4	0.65
2D - 11/20	74.6	17.6	0.015
2E - 11/20	75.6	3.1	0.66
Limestone A	10.7	75	--
Limestone B	16.2	66.6	--
SCU Slurry	8.0	70.0	--
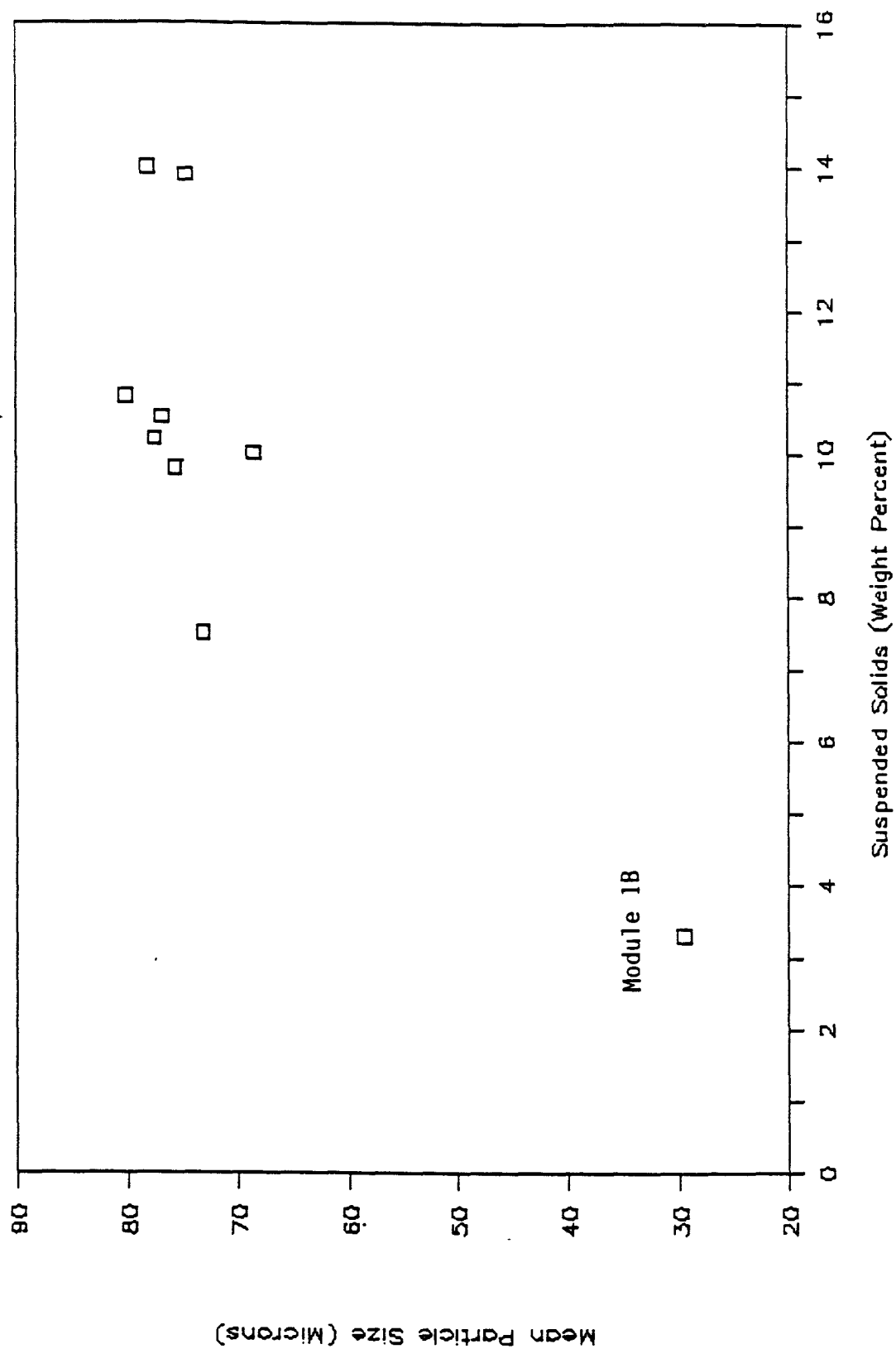
Figure 6-1. Mean Particle Size vs. Slurry Suspended Solids

IP12_006382
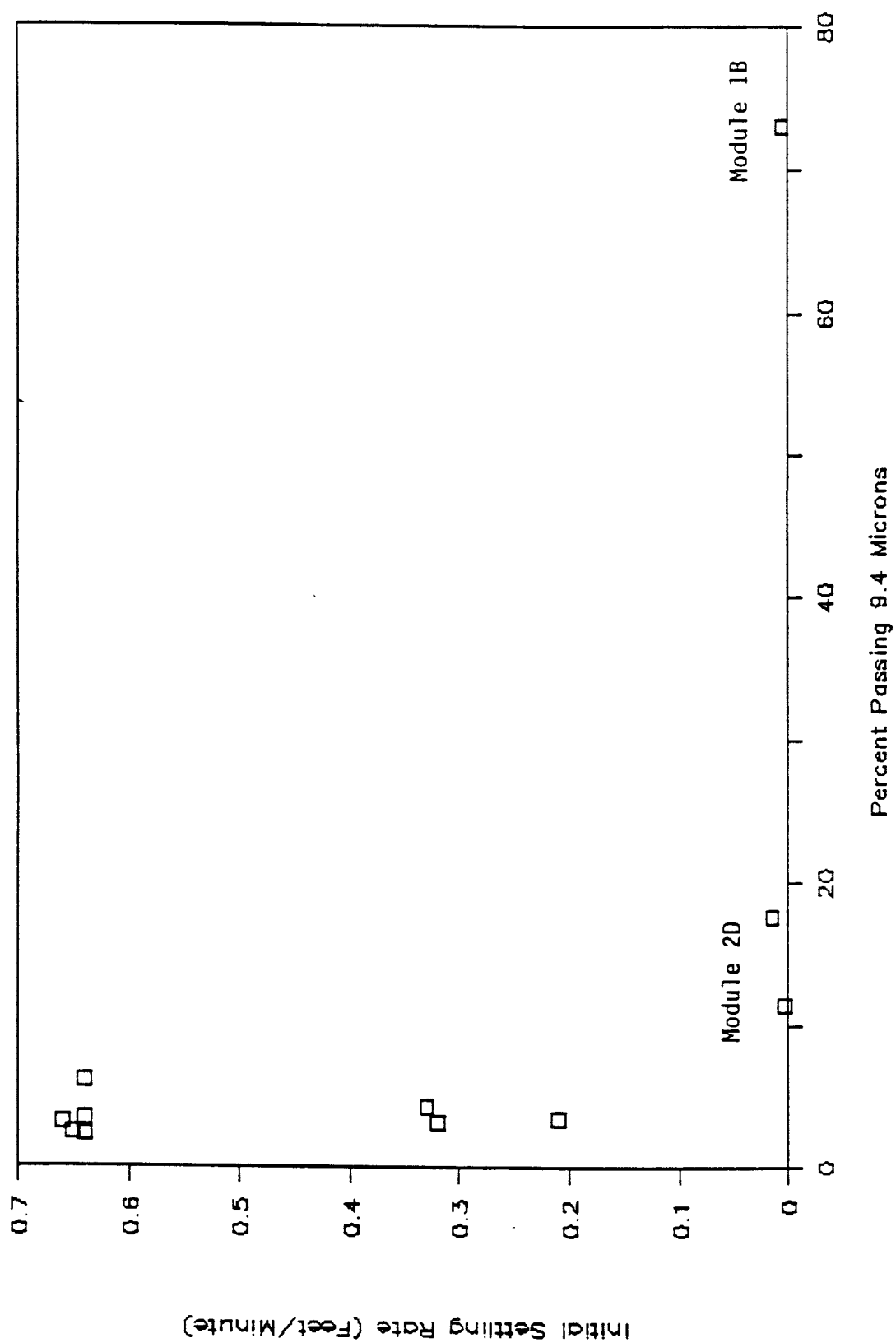
Figure 6-2. Settling Rate vs. Percentage of Fines

IP12_006383

Section 7

DISCUSSION

The current study was initiated to investigate the cause of two scrubber
system operating problems encountered by IPP: nozzle blockage due to scaling
in the reaction tank and spray headers, and occasional poor solids dewatering
properties that affect thickener and filter operations. While scaling con-
ditions were apparently not prevalent during the November site visit, some
aspects of the results presented in the previous sections appeared to be
related to these operating problems.

MODULE START-UP

Results from analyses of recycle slurry from module 1B, which had recently
been restarted after cleaning, showed that absorbed SO_2 was not being oxi-
dized. The solids in this module were a mixture of limestone and calcium
sulfite/sulfate solid solution. Most of the other scrubber modules were
producing exclusively gypsum solids. The high pH that was prevalent because
of the large excess of limestone in the start-up module appeared to inhibit
oxidation.

At some time, about 24 to 36 hours after start-up, the initial limestone
charge in a start-up module will be depleted, and the pH will drop relatively
rapidly to the normal operating setpoint. It is likely that little or no gyp-
sum solids will have been produced in the module prior to this time. When the
pH drops to the normal operating level (5.7 to 5.9), sulfite oxidation will
increase to its normal level near 100%. The probable result will be gypsum

7-1

scale formation on surfaces in contact with the recycle slurry as gypsum is produced without adequate seed crystals to desupersaturate the liquor. Eventually, gypsum solids will accumulate in the reaction tank, and the gypsum relative saturation will fall below the scaling threshold. Gypsum will continue to slowly accumulate on surfaces that scaled during the transition period, however.

Scaling conditions in a start-up module will also be promoted by use of recovered water to fill the start-up module reaction tank. This water has an initially high gypsum relative saturation. When excess limestone is added, the relative saturation will increase as the liquid-phase calcium concentration increases from limestone dissolution. Under these conditions, the gypsum scaling threshold may be exceeded even before the module is operated.

Due to the likelihood that the module start-up conditions are contributing to scaling, it is recommended that a modified start-up procedure be developed and tested. Seed crystals from another module should be used instead of excess limestone to build up solids in the start-up module reaction tank. At least 20% cooling tower blowdown should be added to the tank in addition to recovered water. This should reduce the initial gypsum relative saturation.

IPP had questioned whether changing the start-up procedure would reduce scaling in the scrubber modules if existing scale was not completely removed before start-up. At this time, the relative contribution of start-up conditions to the overall problem is unknown and can be evaluated only by testing the revised start-up procedure. The benefit of complete scale removal should also be evaluated if this is possible without damaging tank or header lining materials.

7-2

IP12_006385

PROCESS CONTROL

Slurry solids content and pH are the two major chemical process parameters that are actively controlled at IPP. Solids content is controlled by periodic adjustment of the rate at which recovered water is added to the reaction tank. The normal slurry solids content is about 10 wt.%. Slurry pH is controlled by adding limestone in proportion to a feed-forward signal based on boiler load and inlet SO_2 concentration and/or a feed-back signal based on deviation from the slurry pH setpoint. The normal slurry pH setpoint is about 5.8.

During the limited sampling period described in this report, the measured slurry pHs were at or below 6.0 for all modules except 1B, which was in a start-up condition, and 2D, to which excess limestone had been added to increase the solids content. The on-line pH meters were operating well. Limestone utilization was very good at pH levels up to about 6.0.

IPP personnel have stated that the pH setpoint is occasionally raised in modules that are not meeting the required SO_2 removal efficiency. Based on results discussed above, it is possible that this operating strategy may be counterproductive. Extended operation of a module at a pH higher than about 6.0 will produce calcium sulfite solids and decrease limestone utilization. Both of these results will decrease the gypsum content of the slurry and will make the solids more difficult to dewater. If the setpoint is then returned to normal, a scaling episode may occur before the solids composition in the reaction tank returns to normal. Once scale is formed on a surface, it will continue to grow even when operation returns to normal.

To maintain good limestone utilization and high sulfite oxidation, an upper limit should be placed on the pH set point. Data from EPRI's High Sulfur Test Center have shown that limestone utilization typically decreases gradually with increasing slurry pH until an operating pH limit is reached above which a very rapid decrease in utilization occurs with only a slight further increase

7-3

in pH. The actual pH level above which this rapid decrease in utilization occurs will vary from system to system, depending on hold tank size, limestone characteristics, and system chemistry. The limited data obtained at IPP during this study suggest that pH 6.0 would be a conservative upper limit for the set point. Good utilization and oxidation were observed in modules operating below pH 6.0.

This suggested upper limit of pH 6.0 is only an approximation. Data from Module 2D, to which excess limestone was added to increase the solids content (pH 6.25, 78% utilization), and Module 1B, which was in a start-up mode (pH 6.39, 16% utilization), do show, however, that the expected sharp decrease in utilization occurs at some pH above about 6.2. The use of pH 6.0 as an upper limit for the set point should provide a comfortable margin to maintain good utilization and high oxidation while allowing for some error in the pH monitors.

Normally, operating modules were meeting emission limits while operating at pH levels from 5.7 to 5.9. If an operating pH higher than 6.0 is required to maintain compliance, some problem with the scrubber, such as nozzle plugging, is indicated. Options other than increasing the pH to maintain compliance appear to be limited to operating the spare module so that the nozzles in the problem module can be examined and cleaned if necessary. If this option is not feasible due to the frequency of the occurrence of this problem, consideration might be given to intermittent use of a mass transfer additive such as DBA.

The slurry solids contents measured during the Codan/Radian site visit ranged from 7.5% in module 1A to 10.8% in module 2B (not including modules 1B and 2D). It was determined that the IPP lab method used for slurry solids control was consistently overestimating the slurry solids contents. Examination of previous laboratory results showed that the estimated solids contents were

occasionally as low as 6% to 7% in non-start-up modules. The actual solids contents may have been even lower. Since a low slurry solids content can cause, or at least contribute, to the severity of a scaling episode, it is possible that low-solids excursions are part of the overall problem. It is suggested that, in addition to correcting the overestimation of the slurry solids content, the control point for suspended solids concentration should be increased to about 15 wt.% solids.

The IPP laboratory method for estimating slurry solids content is based on a correlation between slurry density and solids content. This correlation was developed using the slower but more accurate gravimetric method. This method should be adequate for controlling the solids content provided that some precautions are taken to obtain sufficiently accurate results. Detailed procedures for determining slurry solids content based on slurry density measurements are described in EPRI Report CS-3612, FGD Chemistry and Analytical Methods Handbook, Volume 2. Copies of the appropriate methods are presented in Appendix B of this report.

In the EPRI method, the slurry solids content versus slurry density calibration curve is calculated using measured solids and liquor specific gravities. The solids specific gravity is unlikely to change significantly for a given system, but the liquor specific gravity may change and should be checked occasionally.

Appendix A

SCANNING ELECTRON PHOTOMICROGRAPHS AND
PARTICLE SIZE DISTRIBUTIONS DETERMINED BY
MICROTRAC PARTICLE SIZE ANALYZER

```
                                    :ch-top  %pass  %-ch      :  summary data
                                    :300.00  100.0   0.2      :  dv:    0.8073
                                    :212.00   99.8   3.9      :  %16:   32.09
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9150.00   96.0  16.4      :  %50:   71.47
:MICROTRAC DATA: DATA COLLECTION    :106.00   79.6  26.3      :  %84:  117.87
:                                   : 75.00   53.3  20.4      :  mv:    73.14
:sequence #:       3 record #:     3: 53.00   32.9  12.7      :  cs:     0.185
:sample date/time:  11/29/90 20:22 : 38.00   20.2   7.7      :
:sample ident: MOD 1A              : 27.00   12.4   4.0      :  sd:    42.88
:sample    ID :  1:15 PM,11/19/90  : 19.00    8.5   2.5      :  ma:    32.38
:lot       code: CODAN1 / JIM WILHELM: 13.00   6.0   1.8      LMMMMMMMMMMMMMMMMr
:account  #:   FGD GYPSUM          :  9.40    4.2   0.6      :  parameters
:source: 7995 1.6-300. SRA         :  6.60    3.5   0.3      :  name     value
:length: 200 sec.    run #: 100/   :  4.70    3.2   1.4      :smpl amt
:sample preparation code:          :  3.30    1.8   1.1      :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA :  2.40    0.8   0.8      :disp amt
: SONIC BATH                       :                         :disp med
:                                  :                         :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                       :agitat'n
:                                  :                         :agit tim
:                                  :                         :circul'n
:F6-auto abort         F4-remote   :                         :circ tim
:                                  :                         :param #1
:                                  :                         :param #2
:                                  :                         :param #3
```

```
                                    :ch-top  %pass  %-ch      :   summary data
                                    :300.00  100.0   1.0      :  dv:    0.9015
                                    :212.00   99.0   5.3      :  %16:   32.37
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9150.00   93.1  14.2      :  %50:   63.73
:MICROTRAC DATA: DATA COLLECTION     :106.00   78.9  24.0     :  %84:  121.37
:                                    :  75.00  54.9  20.7     :  mv:    75.01
:sequence #:      6 record #:      6: 53.00   34.2  13.5      :  cs:     0.165
:sample date/time:  11/29/90 20:47 : 38.00   20.7   9.2      :
:sample ident: MOD 2A                : 27.00   11.5   3.3     :  sd:    44.80
:sample    ID :  3:45 PM,11/19/90    : 19.00    7.6   2.2     :  ma:    36.28
:lot       code: CODAN1 / JIM WILHELM: 13.00   5.4   2.0     LMMMMMMMMMMMMMMMM
:account  #:    FGD GYPSUM           :  9.40   3.4   0.8      :   parameters
:source: 7995 1.6-300. SRA           :  6.60   2.6   0.4      :  name      value
:length: 200 sec.    run #: 100/     :  4.70   2.1   1.1      :smpl amt
:sample preparation code:            :  3.30   1.1   0.7      :disprsnt
:notes:   ISOPROPYL ALCOHOL + ULTRA  :  2.40   0.3   0.3      :disp amt
:  SONIC BATH                        :                        :disp med
:                                    :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                         :agitat'n
:                                    :                        :agit tim
:                                    :                        :circul'n
:F6-auto abort          F4-remote    :                        :circ tim
:                                    :                        :param #1
:                                    :                        :param #2
:                                    :                        :param #3


                                    :ch-top  %pass  %-ch      :   summary data
                                    :300.00  100.0   4.5      :  dv:    0.2640
                                    :212.00   95.5   2.4      :  %16:    2.80
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9150.00   93.1   1.6      :  %50:    6.57
:MICROTRAC DATA: DATA COLLECTION     :106.00   91.5   4.5     :  %84:   42.79
:                                    :  75.00  87.0   1.7     :  mv:    23.46
:sequence #:      9 record #:      9: 53.00   85.3   1.9      :  cs:     1.162
:sample date/time:  11/29/90 21:06 : 38.00   83.4   2.3      :
:sample ident: MOD 1B                : 27.00   81.1   3.4     :  sd:    20.00
:sample    ID :  1:32 PM,11/19/90    : 19.00   77.7   5.3     :  ma:     5.16
:lot       code: CODAN1 / JIM WILHELM: 13.00   71.8   8.1     LMMMMMMMMMMMMMMMM
:account  #:    FGD GYPSUM           :  9.40   63.6  13.5     :   parameters
:source: 7995 1.6-300. SRA           :  6.60   50.1  10.7     :  name      value
:length: 200 sec.    run #: 100/     :  4.70   39.5  16.2     :smpl amt
:sample preparation code:            :  3.30   23.3  13.1     :disprsnt
:notes:   ISOPROPYL ALCOHOL + ULTRA  :  2.40   10.2  10.2     :disp amt
:  SONIC BATH                        :                        :disp med
:                                    :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                         :agitat'n
:                                    :                        :agit tim
:                                    :                        :circul'n
:F6-auto abort          F4-remote    :                        :circ tim
:                                    :                        :param #1
:                                    :                        :param #2
:                                    :                        :param #3
```

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```
                                    :ch-top  %pass  %-ch        :   summary data
                                    :300.00  100.0   1.0        :  dv:    0.7463
                                    :212.00   99.0   5.3        :  %16:  38.30
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9:150.00  93.2  16.3   :  %50:  77.15
:MICROTRAC DATA: DATA COLLECTION    :106.00   76.3  28.3       :  %84: 125.25
:                                   : 75.00   48.0  22.5       :  mv:   80.08
:sequence #:     12 record #:    12: 53.00   25.5   9.7        :  cs:    0.145
:sample date/time:  11/29/90 21:25 : 38.00   15.8   6.4        :
:sample ident: MOD 2B              : 27.00    9.4   3.4        :  sd:   43.47
:sample   ID :  3:59 PM,11/19/90   : 19.00    6.0   2.4        :  ma:   41.46
:lot      code: CODAN1 / JIM WILHELM: 13.00    3.6   1.3       LMMMMMMMMMMMMMMMM!
:account  #:    FGD GYPSUM         :  9.40    2.3   0.1        :   parameters
:source: 7995 1.6-300. SRA         :  6.60    2.2   0.3        :   name    value
:length: 200 sec.   run #: 100/    :  4.70    1.3   0.9        :smpl amt
:sample preparation code:          :  3.30    1.0   0.6        :disprsnt
:notes:   ISOPROPYL ALCOHOL + ULTRA :  2.40    0.3   0.3       :disp amt
: SONIC BATH                       :                           :disp med
:                                  :                           :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                       :agitat'n
:                                  :                           :agit tim
:                                  :                           :circul'n
:F6-auto abort       F4-remote     :                           :circ tim
:                                  :                           :param #1
:                                  :                           :param #2
:                                  :                           :param #3
```

```
                                  :ch-top  %pass  %-ch      :   summary data
                                  :300.00  100.0   0.0      :   dv:   0.5388
                                  :212.00  100.0   3.9      :   %16:   37.33
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3150.00   96.1  18.3   :   %50:   76.12
:MICROTRAC DATA: DATA COLLECTION  :106.00   77.8  28.9     :   %84:  120.89
:                                 : 75.00   49.0  21.0     :   mv:   76.79
:sequence #:     15 record #:    15: 53.00   27.9  11.5    :   cs:    0.144
:sample date/time:  12/02/90 11:38 : 38.00   16.5   7.3    :
:sample ident: MOD 2B             : 27.00    9.2   3.8     :   sd:   41.78
:sample    ID :  LOW LOAD,11/20/90 : 19.00    5.4   1.7    :   ma:   41.55
:lot      code: CODAN1 / JIM WILHELM: 13.00    3.7   1.3   LMMMMMMMMMMMMMMMMMMMM
:account  #:   FGD GYPSUM         :  9.40    2.4   0.7     :   parameters
:source: 7395 1.6-300. SRA        :  6.60    1.7   0.0     :   name     value
:length: 200 sec.   run #: 100/   :  4.70    1.7   0.8     :smpl amt
:sample preparation code:         :  3.30    0.9   0.6     :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA :  2.40    0.4   0.4    :disp amt
: SONIC BATH                      :                        :disp med
:                                 :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                     :agitat'n
:                                 :                        :agit tim
:                                 :                        :circul'n
:F6-auto abort         F4-remote  :                        :circ tim
:                                 :                        :param #1
:                                 :                        :param #2
:                                 :                        :param #3
```

```
                                  :ch-top  %pass  %-ch      :   summary data
                                  :300.00  100.0   2.5      :   dv:   0.8632
                                  :212.00   97.5  10.0      :   %16:   15.83
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3150.00   87.5  16.1   :   %50:   71.30
:MICROTRAC DATA: DATA COLLECTION  :106.00   71.5  19.0     :   %84:  140.41
:                                 : 75.00   52.4  14.3     :   mv:   77.99
:sequence #:     18 record #:    18: 53.00   38.1   9.3    :   cs:    0.292
:sample date/time:  12/02/90 12:10 : 38.00   28.8   6.7    :
:sample ident: MOD 2D             : 27.00   22.1   4.0     :   sd:   62.29
:sample    ID : 11/19/90          : 19.00   18.0   3.3     :   ma:   20.55
:lot      code: CODAN1 / JIM WILHELM: 13.00   14.2   2.8   LMMMMMMMMMMMMMMMMMMMM
:account  #:   FGD GYPSUM         :  9.40   11.4   2.1     :   parameters
:source: 7395 1.6-300. SRA        :  6.60    3.3   2.3     :   name     value
:length: 200 sec.   run #: 100/   :  4.70    7.0   3.2     :smpl amt
:sample preparation code:         :  3.30    3.8   2.3     :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA :  2.40    1.5   1.5    :disp amt
: SONIC BATH                      :                        :disp med
:                                 :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                     :agitat'n
:                                 :                        :agit tim
:                                 :                        :circul'n
:F6-auto abort         F4-remote  :                        :circ tim
:                                 :                        :param #1
:                                 :                        :param #2
:                                 :                        :param #3
```

```
                                    :ch-top %pass %-ch         :  summary data
                                    :300.00 100.0  3.3         :  dv:   0.4120
                                    :212.00  96.7  8.5         :  %16:   8.40
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3 :150.00  88.2 15.0        :  %50:  66.73
:MICROTRAC DATA: DATA COLLECTION     :106.00  73.2 17.9        :  %84: 137.72
:                                    : 75.00  55.3 13.9        :  mv:   74.55
:sequence #:     24 record #:    24: 53.00  41.3  8.0         :  cs:    0.367
:sample date/time:  12/02/90 12:42 : 38.00  33.3  7.2         :
:sample ident: MOD 2D LOW LOAD     : 27.00  26.1  5.0         :  sd:   64.66
:sample    ID : 11/20/90           : 19.00  21.1  1.8         :  ma:   16.35
:lot      code: CODAN1 / JIM WILHELM: 13.00  19.3  1.7        LMMMMMMMMMMMMMMMMM
:account  #:   FGD GYPSUM           :  9.40  17.6  4.3        :   parameters
:source: 7995 1.6-300. SRA          :  6.60  13.3  3.5        :  name     value
:length: 200 sec.   run #: 100/     :  4.70   3.8  4.4        :smpl amt
:sample preparation code:           :  3.30   5.4  3.3        :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA  :  2.40   2.1  2.1        :disp amt
: SONIC BATH                        :                         :disp med
:                                   :                         :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                       :agitat'n
:                                   :                         :agit tim
:                                   :                         :circul'n
:F6-auto abort        F4-remote     :                         :circ tim
:                                   :                         :param #1
:                                   :                         :param #2
:                                   :                         :param #3
```

```
                                    :ch-top %pass %-ch         :  summary data
                                    :300.00 100.0  0.7         :  dv:   0.9011
                                    :212.00  99.3  3.2         :  %16:  30.30
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3 :150.00  96.1 10.0        :  %50:  66.11
:MICROTRAC DATA: DATA COLLECTION     :106.00  86.1 25.5        :  %84: 103.44
:                                    : 75.00  60.6 26.3        :  mv:   68.53
:sequence #:     27 record #:    27: 53.00  34.4 13.8         :  cs:    0.213
:sample date/time:  12/02/90 13:03 : 38.00  20.6  7.1         :
:sample ident: MOD 1E 1:50 PM      : 27.00  13.5  4.3         :  sd:   36.27
:sample    ID : 11/13/90           : 19.00   9.2  1.9         :  ma:   28.10
:lot      code: CODAN1 / JIM WILHELM: 13.00   7.3  1.3        LMMMMMMMMMMMMMMMMM
:account  #:   FGD GYPSUM           :  9.40   6.0  1.2        :   parameters
:source: 7995 1.6-300. SRA          :  6.60   4.8  0.5        :  name     value
:length: 200 sec.   run #: 100/     :  4.70   4.3  1.8        :smpl amt
:sample preparation code:           :  3.30   2.5  1.4        :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA  :  2.40   1.1  1.1        :disp amt
: SONIC BATH                        :                         :disp med
:                                   :                         :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                       :agitat'n
:                                   :                         :agit tim
:                                   :                         :circul'n
:F6-auto abort        F4-remote     :                         :circ tim
:                                   :                         :param #1
:                                   :                         :param #2
:                                   :                         :param #3
```

:ch-top %pass %-ch   : summary data
:300.00 100.0 0.3   : dv: 1.0151
:212.00 99.2 5.7   : %16: 33.34

IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9150.00 93.5 14.1 : %50: 71.56
:MICROTRAC DATA: DATA COLLECTION :106.00 79.4 25.8 : %84: 120.40
: : 75.00 53.5 22.7 : mv: 75.64
:sequence #: 30 record #: 30: 53.00 30.3 11.5 : cs: 0.174
:sample date/time: 12/02/90 13:25 : 38.00 19.3 7.8 :
:sample ident: MOD 2E 4:30 PM : 27.00 11.5 4.2 : sd: 43.53
:sample ID : 11/13/90 : 19.00 7.3 2.5 : ma: 34.56
:lot code: CODAN1 / JIM WILHELM: 13.00 4.8 1.7 LMMMMMMMMMMMMMMMMMM
:account #: FGD GYPSUM : 9.40 3.1 0.2 : parameters
:source: 7995 1.6-300. SRA : 6.60 2.3 0.0 : name value
:length: 200 sec. run #: 100/ : 4.70 2.3 1.2 :smpl amt
:sample preparation code: : 3.30 1.7 0.9 :disprsnt
:notes: ISOPROPYL ALCOHOL + ULTRA : 2.40 0.8 0.8 :disp amt
: SONIC BATH : :disp med
: : :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9 :agitat'n
: : :agit tim
: : :circul'n
:F6-auto abort F4-remote : :circ tim
: : :param #1
: : :param #2
: : :param #3

:ch-top %pass %-ch   : summary data
:300.00 100.0 0.1   : dv: 1.2733
:212.00 99.3 6.0   : %16: 38.88

IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9150.00 93.8 15.7 : %50: 73.33
:MICROTRAC DATA: DATA COLLECTION :106.00 78.2 26.4 : %84: 122.40
: : 75.00 51.7 22.8 : mv: 77.82
:sequence #: 33 record #: 33: 53.00 28.9 13.7 : cs: 0.109
:sample date/time: 12/02/90 17:03 : 38.00 15.2 8.4 :
:sample ident: MOD 2E LOW LOAD : 27.00 6.8 2.9 : sd: 41.75
:sample ID : 11/20/90 : 19.00 3.9 3.1 : ma: 55.29
:lot code: CODAN1 / JIM WILHELM: 13.00 0.8 0.8 LMMMMMMMMMMMMMMMMMM
:account #: FGD GYPSUM : 9.40 0.0 0.0 : parameters
:source: 7995 1.6-300. SRA : 6.60 0.0 0.0 : name value
:length: 200 sec. run #: 100/ : 4.70 0.0 0.0 :smpl amt
:sample preparation code: : 3.30 0.0 0.0 :disprsnt
:notes: : 2.40 0.0 0.0 :disp amt
: : :disp med
: : :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9 :agitat'n
: : :agit tim
: : :circul'n
:F6-auto abort F4-remote : :circ tim
: : :param #1
: : :param #2
: : :param #3

```
                              :ch-top  %pass  %-ch      :  summary data
                              :300.00  100.0   0.0      :  dv:    0.8351
                              :212.00  100.0   4.3      :  %16:   41.38
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3150.00   95.1  14.6      :  %50:   75.85
:MICROTRAC DATA: DATA COLLECTION     :106.00   80.5  31.4      :  %84:  116.55
:                                    : 75.00   49.1  25.6      :  mv:    77.39
:sequence #:      36  record #:    36: 53.00   23.5   9.7      :  cs:     0.143
:sample date/time:  12/02/30 17:33 : 38.00   13.8   5.2      :
:sample ident: MOD 1F 2:07 PM       : 27.00    8.7   3.2      :  sd:    37.58
:sample    ID : 11/13/30            : 19.00    5.4   1.4      :  ma:    41.87
:lot       code: CODAN1 / JIM WILHELM: 13.00   4.0   0.5      LMMMMMMMMMMMMMMMM
:account  #:    FGD GYPSUM          :  9.40    3.4   1.5      :   parameters
:source: 7995 1.6-300. SRA          :  6.60    2.0   0.2      :  name    value
:length: 200 sec.   run #: 100/     :  4.70    1.7   0.9      :smpl amt
:sample preparation code:           :  3.30    0.8   0.6      :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA  :  2.40    0.2   0.2      :disp amt
: SONIC BATH                        :                        :disp med
:                                   :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                        :agitat'n
:                                   :                        :agit tim
:                                   :                        :circul'n
:F6-auto abort         F4-remote    :                        :circ tim
:                                   :                        :param #1
:                                   :                        :param #2
:                                   :                        :param #3


                              :ch-top  %pass  %-ch      :  summary data
                              :300.00  100.0   0.0      :  dv:    0.1350
                              :212.00  100.0   0.0      :  %16:    2.33
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3150.00  100.0   0.0      :  %50:    4.20
:MICROTRAC DATA: DATA COLLECTION     :106.00  100.0   2.0      :  %84:   17.38
:                                    : 75.00   98.0   3.0      :  mv:    10.63
:sequence #:      39  record #:    39: 53.00   95.1   1.7      :  cs:     1.514
:sample date/time:  12/02/30 17:59 : 38.00   93.4   3.7      :
:sample ident: A SLURRY TANK UNIT 1: 27.00   89.7   5.0      :  sd:     7.82
:sample    ID : L/S SLURRY          : 19.00   84.7   3.9      :  ma:     3.96
:lot       code: CODAN1 / JIM WILHELM: 13.00   80.8   5.8      LMMMMMMMMMMMMMMMM
:account  #:    FGD GYPSUM          :  9.40   75.0   3.9      :   parameters
:source: 7995 1.6-300. SRA          :  6.60   65.1   7.6      :  name    value
:length: 200 sec.   run #: 100/     :  4.70   57.5  21.1      :smpl amt
:sample preparation code:           :  3.30   36.4  13.0      :disprsnt
:notes:  ISOPROPYL ALCOHOL + ULTRA  :  2.40   17.4  17.4      :disp amt
: SONIC BATH                        :                        :disp med
:                                   :                        :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM3                        :agitat'n
:                                   :                        :agit tim
:                                   :                        :circul'n
:F6-auto abort         F4-remote    :                        :circ tim
:                                   :                        :param #1
:                                   :                        :param #2
:                                   :                        :param #3
```

```
                                          :ch-top  %pass  %-ch      :  summary data
                                          :300.00  100.0   0.2      :  dv:     0.1843
                                          :212.00   99.8   0.6      :  %16:    2.46
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9  150.00   99.2   1.1      :  %50:    4.88
:MICROTRAC DATA: DATA COLLECTION          :106.00   98.1   4.0      :  %84:   27.25
:                                         :  75.00   94.1   3.3      :  mv:    16.21
:sequence #:      42  record #:        42: 53.00    90.9   2.7      :  cs:     1.348
:sample date/time:   12/05/90 18:55  :  38.00      88.2   4.3      :
:sample ident: TANK B UNIT 2          :  27.00      83.9   4.1      :  sd:    12.40
:sample     ID : L/S 11/19/90         :  19.00      79.8   5.6      :  ma:     4.45
:lot       code: CODAN1 / JIM WILHELM:  13.00      74.2   7.6      LMMMMMMMMMMMMMMMMMM
:account   #:      FGD GYPSUM          :   9.40     66.6   9.3      :   parameters
:source: 7395 1.6-300. SRA             :   6.60     56.7   7.4      :  name    value
:length: 200 sec.    run #: 100/       :   4.70     49.3  18.1      :smpl amt
:sample preparation code:              :   3.30     31.2  16.3      :disprsnt
:notes:   ISOPROPYL ALCOHOL + ULTRA :     2.40     14.9  14.9      :disp amt
: SONIC BATH                           :                            :disp med
:                                         :                            :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                        :agitat'n
:                                         :                            :agit tim
:                                         :                            :circul'n
:F6-auto abort         F4-remote          :                            :circ tim
:                                         :                            :param #1
:                                         :                            :param #2
:                                         :                            :param #3


                                          :ch-top  %pass  %-ch      :  summary data
                                          :300.00  100.0   0.0      :  dv:     0.2164
                                          :212.00  100.0   0.0      :  %16:    2.68
IMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9  150.00  100.0   0.0      :  %50:    5.79
:MICROTRAC DATA: DATA COLLECTION          :106.00  100.0   0.0      :  %84:   14.33
:                                         :  75.00  100.0   0.0      :  mv:     7.96
:sequence #:      45  record #:        45: 53.00   100.0   0.0      :  cs:     1.306
:sample date/time:   12/05/90 13:36  :  38.00     100.0   1.5      :
:sample ident: THICKENED SCU          :  27.00      98.5   6.9      :  sd:     5.83
:sample     ID : 11/19/90 2:48 PM     :  19.00      91.6   9.8      :  ma:     4.60
:lot       code: CODAN1 / JIM WILHELM:  13.00      81.8  11.8      LMMMMMMMMMMMMMMMMMM
:account   #:      FGD GYPSUM          :   9.40     70.0  15.3      :   parameters
:source: 7395 1.6-300. SRA             :   6.60     54.7  11.0      :  name    value
:length: 200 sec.    run #: 100/       :   4.70     43.7  17.7      :smpl amt
:sample preparation code:              :   3.30     26.0  14.5      :disprsnt
:notes:   ISOPROPYL ALCOHOL + ULTRA :     2.40     11.4  11.4      :disp amt
: SONIC BATH                           :                            :disp med
:                                         :                            :dmed amt
LMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMMM9                        :agitat'n
:                                         :                            :agit tim
:                                         :                            :circul'n
:F6-auto abort         F4-remote          :                            :circ tim
:                                         :                            :param #1
:                                         :                            :param #2
:                                         :                            :param #3
```

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MODULE 1A – 11/19/90 – 480X

MODULE 1B 11/19/90 – 480X

MODULE 1E - 11/19/90 - 480X

MODULE 1F 11/19/90 - 480X

MODULE 2A - 11/19/90 - 480X

MODULE 2B 11/19/90 - 480X

MODULE 2D - 11/19/90 - 480X
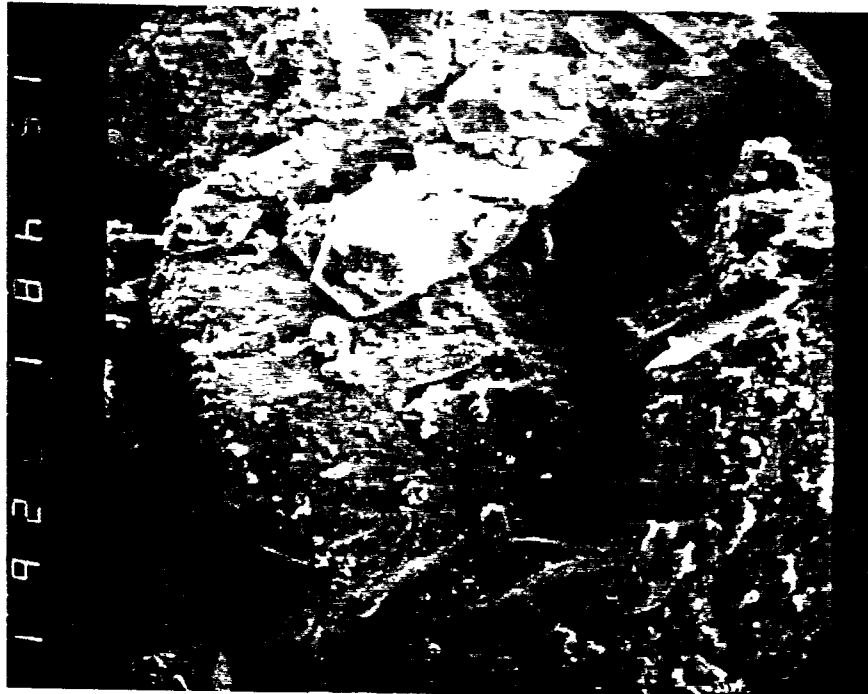
MODULE 2E 11/19/90 - 480X

MODULE 2B - 11/20/90 LOW LOAD - 480X
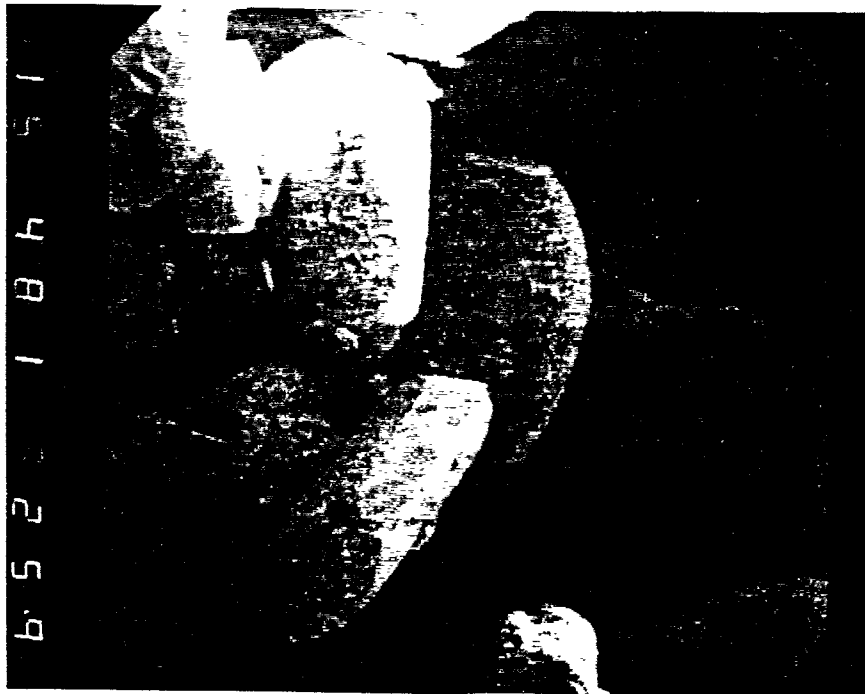
MODULE 2D 11/20/90 LOW LOAD- 480X

MODULE 2E - 11/20/90 LOW LOAD- 480X
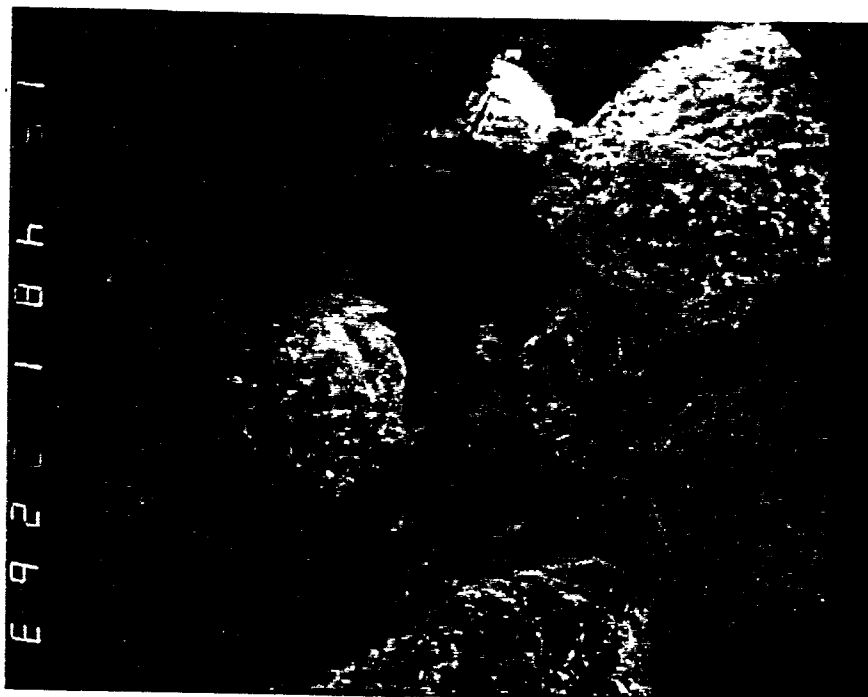
SCU SLURRY 11/19/90 - 1000X

LIMESTONE – SLURRY TANK A 11/19/90 – 1000X
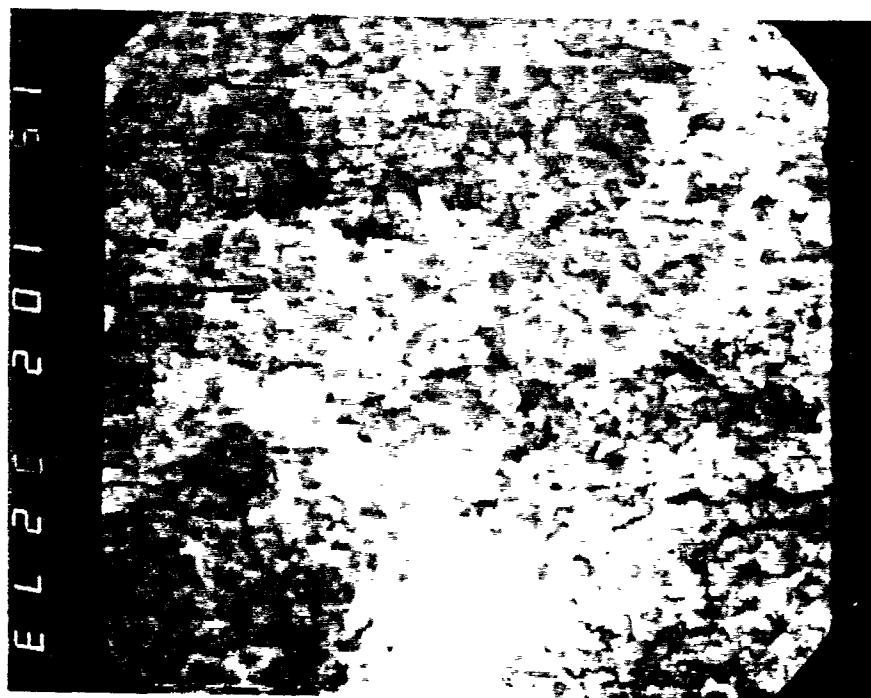
LIMESTONE – SLURRY TANK B 11/19/90 – 1000X

Appendix B

DENSITY MEASUREMENT METHODS

D1 Liquid Phase Density

1.0 METHOD DESCRIPTION

This method consists of filling a clean, dry, weighed, calibrated volumetric flask with liquor of a known temperature, determining the total mass of the flask contents, and dividing that mass by the flask volume. If the volumetric flask is filled to the mark, the specified volume of the flask (e.g., 100.00 mL) is used. If the flask is completely filled with liquid (so there are no air bubbles below the stopper), the volume is determined by calibrating the flask with deionized water.

2.0 SIGNIFICANCE AND USE

Density is a measurement of the mass of a given volume of material. It is usually reported in units of grams per cubic centimeter or grams per milliliter. Because the density of a material changes as it is heated or cooled, temperature is specified when density is reported.

This procedure is used to determine the density of scrubber liquors from lime, limestone, magnesia, dual alkali, or spray drying processes. The density of scrubber liquors is used to calculate weight percent solids by either the Cassia flask density or the constant volume density method. It is also used to calculate dilution factors associated with sample collection and handling.

3.0 RANGE OF PRECISION AND APPLICABILITY

Limit of precision: 0.00012 g/mL (assuming temperature measurements are precise to 0.5°C)

Applicable range of procedure: This procedure is applicable for all temperatures and densities expected for scrubber liquors

4.0   ELAPSED TIME AND LABOR HOURS REQUIRED

Elapsed time from sample receipt to final answer: 3.0 hr

Operator/analyst time: 0.5 hr

Preparation for analysis (drying, weighing flasks): 2.5 hr

Sample handling (weighing, recording): 0.5 hr

5.0   DEFINITIONS

<u>volumetric</u>--involving the precise and accurate measurement of volume, typically using calibrated vessels

<u>Cassia flask</u>--a volumetric flask with a calibrated neck, e.g., a 100 mL flask with markings from 100 to 110 mL graduated in 0.1 mL increments

<u>bias</u>--a systematic error inherent in a procedure that is introduced by the method of sampling or analysis

6.0   INTERFERENCES/SOURCES OF ERROR

The most common source of error is improper sampling. If a liquor sample is obtained that is not representative of the bulk liquor, further analytical work will not deliver useful information. Great care should be taken during sampling to obtain a representative sample of the bulk liquor.

7.0   ALTERNATE METHODS

This is the only method for determining liquor density provided in this handbook. See ASTM Method D-1429 (1) for other methods.

8.0   PRECAUTIONS/CRITICAL STEPS

●   For highest precision, density determinations should be performed in triplicate.

- Temperature readings for deionized water (used for flask calibration) and for liquors should be made to ±0.5°C.

- Flasks should be cleaned at least the day before they will be used and dried in an oven at 100°C overnight. This saves an hour of elapsed time on the day of sampling or analysis.

9.0 EQUIPMENT AND REAGENTS

Apparatus

- Three 100 mL volumetric flasks with stoppers

- Oven for drying flasks, 100°C

- Desiccator

- Toploading balance, accurate to ±0.01 g

- Thermometer (0-80°C)

Reagents

- Source of deionized water (conductivity <5 μmhos/cm)

10.0 QUALITY ASSURANCE/QUALITY CONTROL

Results of this procedure should be verified by performing analyses in triplicate. Check measurement technique by weighing a 100 mL volumetric flask, then filling it to the calibration mark with deionized water and measuring the water temperature. The calculated water density based on a volume of 100.00 mL should be within 0.2% of the value given in Table D1-1 in Section 14.

Results of analysis of two samples of the same material are acceptable if they differ by less than 2% of their average value.

11.0 SAMPLE ACQUISITION AND HANDLING

Samples for liquor density measurements are obtained by filtering a slurry. If the slurry temperature is different from the laboratory temperature, collect the slurry liquor by in-line filtration at the sampling point. Even nonreactive slurries such as limestone feed slurries may exchange material between the liquid and solid phases when the temperature is changed. The conditions for filtering depend upon whether the density measurement is to be made at process

temperature or at laboratory temperature. Reporting requirements for process data will dictate the temperature at which the density must be measured.

Sample Acquisition for Density Measurement at Process Temperature

Collect the liquor by direct in-line filtration at the sampling point. Collect the sample in a clean, dry, weighed, calibrated 100 mL flask that has been marked to identify it and its stopper. Do not dilute the liquor. To minimize temperature changes, immerse the collection flask in a bath at process temperature. (One convenient way to prepare a bath is to fill a small insulated container, for example, a small ice chest, with the slurry being filtered.) When the flask is nearly full of filtrate, measure the temperature of the contents. If the temperature of the flask contents is within 10°C of the slurry temperature, the temperature change will introduce an error of less than 0.5% in the density in most cases. If the temperature of the contents is within 10°C of the process temperature, fill the flask all the way to the top. Replace the stopper, making certain no air bubbles are entrapped. Remove the flask from the temperature bath and take it back to the laboratory. For most streams, the laboratory temperature will be below process temperature and the sample volume will be less after the sample is returned to the laboratory. But, if the flask was filled near process temperature, the laboratory volume does not matter for this measurement.

If the filtered liquor temperature differs from the process temperature by more than 10°C, then the sample must be returned to the laboratory and the temperature returned to within 10°C of process temperature. In this case, fill the flask to above the 100 mL mark, replace the stopper and return the sample to the laboratory.

Sample Acquisition for Density Measurement at Laboratory Temperature

Collect the liquor by direct in-line filtration at the sampling point. Collect the sample in a clean, dry, weighed, calibrated 100 mL flask that has been marked to identify it and its stopper. Do not dilute the liquor. Fill the flask to above the 100 mL mark, replace the stopper and return the sample to the laboratory. In most cases, the sample volume will be less at laboratory temperature than at process temperature so care must be taken to collect enough sample to have at least 100 mL at laboratory temperature.

12.0 STEPWISE PROCEDURE

Flask Calibration

If the flexibility to use either the calibration mark or the total flask volume is needed, then the total volume of each flask will have to be measured. If the flasks are calibrated, then the same flasks may be used for density measurements of scrubber liquors and density or specific gravity measurements of slurries.

1. Label all flasks to be calibrated with permanent identification. Label the flask stopper with the same identification so that each flask will have its own unique stopper. Once labeled, never mix stoppers and flasks.

2. Clean the flasks and stoppers and rinse well with deionized water. Oven dry flasks at 100°C and place in a desiccator to cool to room temperature.

3. Place each stopper in its respective flask, weigh to the nearest 0.01 g, and record the weight of the empty flask plus stopper.

4. Carefully fill the flask completely with deionized water at laboratory temperature. Record the temperature. Add enough deionized water so that the stopper may be placed in the flask with no air bubbles remaining below the stopper. Wipe the outside of the flask dry, weigh to the nearest 0.01 g, and record the weight.

5. Repeat Steps 2 through 4 until three sets of weights are obtained for each flask being calibrated.

6. After the flask calibration is complete, clean, dry, and store the flasks and stoppers.

Density Measurement (Liquors)

1. Weigh a clean, dry, calibrated volumetric flask and its asso-
    ciated stopper to the nearest 0.01 g. Record the weight.

2. Fill the volumetric flask with liquor. The flask may be
    filled either to the 100 mL mark (precisely) or to the top of
    the flask with no air bubbles trapped below the stopper. (See
    Section 11, Sample Acquisition and Handling for details.)
    Record the sample collection point identification.

3. The next step depends upon the temperature at the time the
    sample was collected and whether the density is to be reported
    at process temperature or at laboratory temperature.

    Case 1: The density is to be reported at process temperature,
    the sample was collected within 10°C of process temperature,
    and the flask was completely filled. Wipe the outside of the
    flask to be sure it is clean and dry and weigh the flask, its
    associated stopper, and the contents to the nearest 0.01 g.
    Record the weight, the process temperature, and the tempera-
    ture at which the sample was collected (to the nearest 0.5°).
    Record that the flask was filled all the way to the stopper.

    Case 2: The density is to be reported at process temperature,
    and the sample was collected at more than 10°C from process
    temperature. Bring the sample to within 10°C of process
    temperature. Adjust the liquor volume to the calibration mark
    by removing liquor with a small pipet (using a bulb) or a long
    eyedropper. Wipe the outside of the flask to be sure it is
    clean and dry and weigh the flask, its associated stopper, and
    the contents to the nearest 0.01 g. Record the weight, the
    process temperature, and the temperature at which the volume
    was adjusted (to the nearest 0.5°). Record that the flask was
    filled to the calibration mark.

NOTE: If a thermostatically controlled water bath is available, it should be used to return the sample to process temperature. If a thermostatically controlled water bath is not available, a convenient way to heat the sample is to place a large beaker (at least 1000 mL and preferably 2000 mL) on a hot plate, fill the beaker full of deionized water, and adjust the heater setting so the water temperature stabilizes near process temperature. The liquor sample can then be suspended in the water bath until the sample temperature reaches the desired value.

Case 3: The density is to be reported at laboratory temperature. Allow the flask and contents to come to laboratory temperature. Measure and record the temperature to the nearest 0.5°. Adjust the liquor volume to the calibration mark by removing liquor with a small pipet (using a bulb) or a long eyedropper. Wipe the outside of the flask to be sure it is clean and dry and weigh the flask, its associated stopper, and the contents to the nearest 0.01 g. Record the weight. Record that the flask was filled to the calibration mark.

13.0   DATA RECORDING FORM

See Figure D1-1 for an example data recording form for flask calibration. An example data recording form for liquor density is shown in Figure D1-2.

14.0   CALCULATIONS

Calculations for flask calibration and for liquid phase density are described in this section. The total volume of volumetric flasks filled all the way to the top with deionized water can be calculated using the following equation:

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Analyst: _____   Date: _____

Flask ID	R Weight of Flask plus Stopper plus Deionized Water (g)	F Weight of Empty Flask plus Stopper (g)	W[1] Weight of Deionized W (g)	T Water Temperature °C	D[2] Water Density (g/mL)	V[3] Full Volume of Flask (mL)

_____

[1]$W = R - F$

[2]D is from Table D1-1

[3]$V = \dfrac{W}{D}$

Figure D1-1. Flask Calibration Data Recording Form

Analyst: _____   Date: _____

Sample ID	Flask ID	S Weight of Liquor Sample plus Flask plus Stopper (g)	F[1] Weight of Empty Flask plus Stopper (g)	C Temperature of Liquor Sample (°C)	L Density of Liquor Sample (g/mL)

---

[1]F may be taken from the "Flask Calibration Data Recording Form" if no label has been added to the flask; otherwise, weigh the flask just before sample collection.

Figure D1-2. Liquid Phase Density Data Recording Form

$$V = \frac{R - F}{D} = \frac{W}{D} \qquad \text{(D1-1)}$$

where: V = flask volume filled all the way to the stopper (mL),

R = weight of flask, stopper, and deionized water (g),

F = weight of empty flask and stopper (g),

W = weight of deionized water (g), and

D = relative density of water at the temperature used for flask calibration (g/mL).

Use the following equation to calculate the density of liquor samples:

$$L = \frac{(S - F)}{A} \qquad \text{(D1-2)}$$

where: L = density of the liquor sample (g/mL),

S = weight of liquor sample, flask, and stopper (taken from the "Density Data Recording Form") (g),

F = weight of empty flask and stopper (may be taken from the "Flask Calibration Data Recording Form" if no label has been added to the flask; otherwise, weigh the flask just before sample collection) (g),

A = volume of liquor sample taken (mL). This number will be 100.00 mL if the flask was filled with sample to the 100.00 mL mark; it will be V, "Full Volume of Flask," from Eq. D1-1 if the flask was filled all the way to the stopper.

15.0   PRECISION

It is reported in ASTM Method D-1429 that results with a precision of 0.005 g/mL can be obtained with this method (1).

16.0   REFERENCES

1. 1977 Annual Book of ASTM Standards, Part 31. Test Methods for Specific Gravity of Water and Brine, Method No. D 1429-76. Philadelphia: American Society for Testing and Materials, 1977.

Table D1-1

RELATIVE DENSITY OF WATER FROM 10 TO 55°C (2)

Temperature °C	Relative Density of Water (g/mL)	Temperature °C	Relative Density of Water (g/mL)
10	0.99973	33	0.99473
11	0.99963	34	0.99440
12	0.99952	35	0.99406
13	0.99940	36	0.99371
14	0.99927	37	0.99336
15	0.99913	38	0.99299
16	0.99897	39	0.99262
17	0.99880	40	0.99224
18	0.99862	41	0.99186
19	0.99843	42	0.99147
20	0.99823	43	0.99107
21	0.99802	44	0.99066
22	0.99780	45	0.99025
23	0.99756	46	0.98982
24	0.99732	47	0.98940
25	0.99707	48	0.98896
26	0.99681	49	0.98852
27	0.99654	50	0.98807
28	0.99626	51	0.98762
29	0.99597	52	0.98715
30	0.99567	53	0.98669
31	0.99537	54	0.98621
32	0.99505	55	0.98573

2.   Chemical Rubber Co. Handbook of Chemistry and Physics. 1967.

3.   1977 Annual Book of ASTM Standards. Part 19. Standard Test Method for
     Specific Gravity of Soils, Method No. D 854-58. Philadelphia: American
     Society for Testing and Materials, 1977.

4.   A.E. Behl. Radian Laboratory Notebook No. 00739. December 1982.

D3 Particle Density of Solids

1.0   DESCRIPTION OF METHOD

Particle density is measured by determining the volume of hexane displaced by a known weight of particulate solids.

2.0   SIGNIFICANCE AND USE

Lime, limestone, magnesia, dual alkali, and spray dryer FGD systems all have solids suspended in a liquid (a slurry) at some point in the process. Reagent feeds are sparingly soluble solids for all calcium-based processes, including the precipitation step in the dual alkali process. All these processes produce a sulfur-containing solid product. The solids occur in slurries as small particles suspended in the liquid phase, in filter cakes from wet FGD processes, or as a dry powder from spray dryers. The particle density of these fine particles is needed to calculate slurry weight percent solids by the Cassia flask or the constant volume density method.

Hexane is used to minimize solids dissolution.

3.0   RANGE OF PRECISION AND APPLICABILITY

This method is applicable to the range of particle densities expected for particulate solids from all FGD systems.

4.0   ELAPSED TIME AND LABOR HOURS REQUIRED

Elapsed time from sample receipt to final answer: 2 hrs

Operator/analyst time: 1 hr

Preparation for analysis: 1.5 hrs

Sample handling (weighing, recording, calculating): 0.5 hr

5.0 DEFINITIONS

density--mass per unit volume

specific gravity--the ratio of the weight in air of a given volume of a material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature

6.0 INTERFERENCES/SOURCES OF ERROR

Solids that might contain hydrated salts, especially calcium sulfate dihydrate (gypsum) or magnesium sulfite hydrates, must be dried very carefully to remove adherent moisture without removing the waters of hydration. The solids should be washed (with a 1:1 mixture of acetone for wet scrubber solids) to remove adherent liquor before drying.

7.0 ALTERNATE METHODS

This is the only method provided in this handbook for determining the particle density of solids.

8.0 WARNINGS/PRECAUTIONS/CRITICAL STEPS

- Air entrained in the solids must be removed by subjecting the solids to reduced pressure. To remove all entrapped air, the sides of the desiccator may be tapped lightly.

- The temperature of the hexane must be controlled to avoid errors in measuring its density.

- Flasks must be clean and completely dry before solids are added.

- If reduced pressure is applied for a long period of time, hexane may boil.

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9.0   EQUIPMENT AND REAGENTS

Apparatus

- Laboratory notebook

- Volumetric flasks, 100 mL (three for each sample and one for hexane only)

- Top-loading balance, accurate to ± 0.01 g

- Oven, for drying flasks

- Vacuum desiccator or bell jar high enough to accommodate 100 mL volumetric flasks in an upright position

- Vacuum pump or aspirator

- Stopcock to control flow to pump or aspirator

- Tubing to connect vacuum vessel to pump or aspirator

- Thermometer

- Water bath with thermostatic control to keep hexane and samples at 25°C

Reagents

- Hexane

10.0   QUALITY ASSURANCE/QUALITY CONTROL

Results of this procedure may be verified by replicate measurements and by measuring the particle density of a sample of known material. For example, pure crystalline calcite can be crushed to a particle size similar to the samples to be tested and the particle density measured and compared to that given in standard reference works. No information is available on the precision or bias of this method. Each laboratory will have to establish acceptable limits based on historical experience.

11.0   SAMPLE ACQUISITION/SAMPLE HANDLING

Collect solid samples from reactive slurries by in-line filtration. Rinse the solids with a 1:1 ratio acetone/water mixture. Dry solids that may contain hydrated species at 60°C. Collect solids from nonreactive slurries using

either in-line filtration or laboratory filtration. Rinse with deionized water or a 1.1 acetone/water mixture, depending on solid phase composition. Dry in an oven at 60°C for solids that may contain hydrated species or at 105°C for lime or limestone.

12.0   STEPWISE PROCEDURE

Slurry Solids Particle Density (or Specific Gravity)

1.   Clean, dry, label, stopper, and weigh three 100 mL volumetric flasks. Record weights on the data recording form in Figure D3-1.

2.   Weigh about 25 g of dried slurry solids to the nearest 0.01 g and transfer quantitatively to a 100 mL volumetric flask with hexane. NOTE: The bulk density of spray dryer solids may be so low that a smaller weight must be taken to avoid filling the flask. Record the solid weight, S. Repeat for each of the three flasks.

3.   Add enough hexane to each flask to cover the solids. The flasks should be one-half to two-thirds full.

4.   To remove entrapped air from the solids, place all flasks, unstoppered, in a desiccator with an air outlet. Attach this outlet to a vacuum pump or aspirator and subject the flask contents to a partial vacuum. Control the rate of air bubble release from the solids by adjusting the vacuum flow rate. Continue reduced air pressure until bubble evolution ceases (15-20 minutes).

5.   Remove the flasks from the desiccator and fill nearly to volume with hexane. Stopper the flasks and place them in a water bath at 25°C for 30 minutes. Adjust the volume to exactly 100.00 mL using hexane that has been kept in the 25°C bath.

6. Remove one flask from the constant temperature bath. Wipe the exterior dry and weigh to the nearest 0.01 g. Record this weight, T, on the data recording form.

7. Repeat Steps 5 and 6 for each sample. Leave flasks in the water bath until ready to weigh.

13.0 DATA RECORDING FORM

Figure D3-1 is a data recording form for particle density measurements.

14.0 CALCULATIONS

Calculate the particle density using the following equation:

$$D = \frac{S}{100 - [(T - S)/H]} \qquad (D3-1)$$

where: D = particle density of solids (g/mL),

S = weight of dry solids added to the flask (g),

T = weight of solids and hexane in the flask filled to the 100 mL mark (g), and

H = density of hexane at 25°C (0.6574 g/mL).

15.0 PRECISION AND BIAS

No precision and bias results were reported for this method.

16.0 REFERENCES

1. 1977 Annual Book of ASTM Standards. Part 4, Standard Method of Testing for Specific Gravity of Soils, Method No. C 854. Philadelphia: American Society for Testing and Materials, 1977.

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Analyst: _____   Analysis Date: _____

Density of Hexane (H) = 0.6574 g/mL

Flask ID	Weight of Flask (g)	Weight of Dry Solids plus Container (g)	Weight of Container (g)	S Weight of Dry Solids (g)	Weight of Flask plus Dry Solids plus Hexane (g)	T Weight of Dry Solids plus Hexane (g)	D Particle Density of Solids (g/mL)

Figure D3-1.   Data Recording Form for Determination of Solids Particle Density

F4 Constant Volume Density Method for Weight Percent Solids

1.0 DESCRIPTION OF METHOD

A container of accurately measured volume is completely filled with a slurry sample. The sample weight and volume are used to calculate slurry density. Weight percent solids in the slurry is found from a previously prepared graph of slurry density versus weight percent solids. The graph is calculated using measured values of slurry liquor density and solid particle density. The relationship is not linear. This handbook entry includes instructions for preparing the graph.

2.0 SIGNIFICANCE AND USE

Weight percent solids measurements are commonly used to control slurry blowdown or bleed rate to the thickener. Thickener overflow or underflow may also be checked to monitor operation.

The constant volume density method is widely used to measure weight percent solids. It is the fastest and easiest method, but users have reported that improper application of the method produces significantly biased results. Laboratory investigations have confirmed that even with proper sampling procedures and accurate liquor and solids densities, recoveries with this method are 3% to 4% high for slurries containing 5 and 10 weight percent solids, respectively.

3.0 DETECTION LIMIT AND RANGE OF METHOD

Detection limit: 5% by weight

Applicable range of procedure: 5 to 20 weight percent solids; above 22 weight percent solids, the relationship between slurry density and weight percent solids becomes less reliable

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4.0 ELAPSED TIME AND LABOR HOURS REQUIRED

Elapsed time from sample receipt to final answer: 2.2 hr (Elapsed time is reduced to 0.1 hr if an appropriate curve has already been generated.)

Operator/analyst time: 2.2 hr

Preparation for analysis (curve generation, density determinations): 2.1 hr

Sample handling (collection and weighing): 0.1 hr

5.0 DEFINITIONS

density—mass per unit volume, usually reported as grams/cubic centimeter

specific gravity—weight of a given volume of a substance compared to the weight of an equal volume of water

6.0 SOURCES OF ERROR

The density of slurry liquor varies with the dissolved solids content. Separate graphs of slurry density versus weight percent solids must be prepared for slurries that have liquid phases with varying densities (dissolved solids levels in the liquor).

Temperature variations in the 23°C to 50°C range do not significantly affect slurry density. A maximum variation in density of 1% can be expected.

Sample flasks should be completely filled so that no air space remains. Entrapped air results in a low slurry weight measurement. Allowing the sample flask to overflow also causes error, because incoming slurry displaces liquor more readily than it displaces solids.

7.0   ALTERNATE METHODS

This handbook includes six methods for weight percent solids determination. The choice depends on the concentration of solids in the slurry, the time required to perform the method, and the precision required. These elements are summarized in Table F4-1.

One of the gravimetric methods is specifically applicable to spray dryer and lime feed slurries, while the other is applicable to lime, limestone, and magnesia scrubber slurries. The two gravimetric methods are the most time consuming, but they have the best precision and are the least biased. They should be viewed as referee methods and should be used to validate results of other methods if they are not used for routine measurements.

Two of the alternate methods are based on measuring slurry density (weighing a known volume of slurry). Although these methods are simple and require very little time, they are subject to significant error if the liquor density changes significantly and is not measured. For example for the constant volume density method, if the solid phase density is 2.607 g/mL and the liquid phase density is 0.9986 g/mL, a slurry density of 1.101 g/mL will yield a reading of 15.0 weight percent solids. For the same slurry density and solid phase density and a liquid phase density of 1.020 g/mL, the weight percent solids will be 12.2 weight percent. If the wrong correlation line is used, there will be an absolute difference of 2.8 in the weight percent solids measurement. The precision of the slurry density measurement methods is almost as good as for the gravimetric methods, and once the densities have been determined, these methods can provide weight percent solids data within a few minutes of sample collection.

The centrifuge method is equally simple and fast, but the precision is significantly lower. In addition, application of the centrifuge method requires that a correlation factor be developed by comparison with results obtained using a gravimetric method. The Ohaus moisture balance method, specifically applicable to lime and spray dryer slurries, is precise and fast but can have significant bias for lime slurries.

The nuclear density meter is a widely used on-line instrumental method for weight percent solids determination. Users report varying satisfaction with

Table F4-1

SLURRY WEIGHT PERCENT SOLIDS METHODS COMPARISON

Method	Range (weight percent)	Sources of Bias	Applications	Preparation Time (hr)	Sample Analysis Time (hr/single sample)	Single Operator Coefficient of Variation (%)	Special Equipment Requirements
Gravimetric	1-50	Excessive drying temperature or drying time will cause dehydration and/or decomposition, resulting in negative bias. Reaction of lime slurries with atmospheric CO_2.	Reagent feed (lime, limestone) and circulating slurries, all FGD systems and spray dryers	0.25	2.25	limestone feed 0.50, lime feed .97, limestone scrubber 0.87, lime scrubber 1.5	None
Gravimetric-- SD	15-40	Excessive drying temperature or drying time will cause dehydration and/or decomposition, resulting in negative bias. Reaction of lime slurries with atmospheric CO_2. Solids dissolution by washing.	Spray dryer feed slurries and lime slurries	0.25	4.5 - 13	Spray dryer feed slurries 0.24, lime slurries 0.51	None
Cassia Flask Density	2.5-30	Must be calibrated with a gravimetric method. New calibration required when liquor TDS changes. Collection flask must be filled without overflowing and with no air bubbles.	Reagent feed and circulating slurries, all FGD systems	0.7	0.3	limestone feed 1.6, lime feed 2.1, limestone scrubber 1.8-2.3, lime scrubber 6.7	Cassia flasks
Constant Volume Density	5-22	Must be calibrated with a gravimetric method. New calibration required when liquor TDS changes. Collection flask must be filled without overflowing and with no air bubbles.	Reagent feed and circulating slurries, all FGD systems	2.1	2.2	< 1%	
Centrifuge	0.5-55	Correction factors must be developed for each slurry type based on comparison with gravimetric method	Reagent feed and circulating slurries, all FGD systems	0.10	0.25	limestone feed 4.2, lime feed 6.2, limestone scrubber 4.9, lime scrubber 5.0	Centrifuge

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SLURRY WEIGHT PERCENT SOLIDS METHODS COMPARISON

Method	Range (weight percent)	Sources of Bias	Applications	Preparation Time[1] (hr)	Sample Analysis Time[2] (hr/ single sample)	Single-Operator Coefficient of Variation[3] (%)	Special Equipment Requirements
Ohaus Moisture Balance		High TDS, reactions of lime slurries with atmospheric CO_2	Spray dryer feed slurries and lime slurries	0.05	0.35 0.55	Spray dryer feed slurries 1.2 Lime slurries 2.0	Ohaus moisture balance

[1]Includes labor hours for equipment set-up

[2]Elapsed time from receipt of one sample to calculated final result, including sample preparation, analysis, and calculations. Clean up time is not included. Actual labor hours are less than elapsed time in some cases. Labor hours per sample will be reduced if more than one sample is analyzed.

[3]Coefficient of variation (CV) = (standard deviation/mean) x 100

$$CV = \frac{[\{\Sigma (X_i - \overline{X})^2\}/(n - 1)]^{1/2}}{\overline{X}} \times 100$$

where: X_i = individual measurement
        n = number of replicates
        $\overline{X}$ = mean of measurements

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nuclear density meters, citing calibration difficulties and inaccuracy for low weight percent solids applications.

8.0   PRECAUTIONS/CRITICAL STEPS

- Perform density determinations for solids and liquors in triplicate.

- Prepare a new curve for each slurry of different liquor density. Although changes in either liquor or solids densities will affect the final results, liquor densities are more likely to change than solids densities.

9.0   EQUIPMENT AND REAGENTS

Apparatus

- Linear graph paper

- Ten 100 mL volumetric flasks

- Toploading balance, capable of weighing to 0.01 g

- Thermometer (0-100°C)

- Oven, for drying flasks

- Desiccator with air vent

- Vacuum pump

- Spatula

- Weigh boats

- Constant temperature (water) bath, 25°C

Reagents

- Reagent grade hexane, for solid particle density determination

10.0   QUALITY ASSURANCE/QUALITY CONTROL

Verify weight percent solids values obtained from the constant volume density method by performing replicate analyses on at least 10% of the scrubber slurry samples and by comparing the results with those obtained using a gravimetric method.

11.0   SAMPLE ACQUISITION AND HANDLING

Collect the slurry samples for weight percent solids determinations in clean, dry, labeled, weighed, calibrated, and stoppered 100 mL flasks. The sample collector should wear gloves. Open the sample port and allow it to flush 20-30 seconds before collecting the sample. Fill the sample container completely, but do not allow it to overflow. The incoming slurry displaces liquor more readily than it displaces solids, leading to a biased weight percent solids determination.

12.0   STEPWISE PROCEDURE

Several preliminary steps must be completed before a correlation curve (slurry density versus weight percent solids) can be prepared. First, calibrate several 100 mL flasks. Then, measure the density of slurry solids (1, 2) and the density of slurry liquor. Follow the calculations in Section 14 to prepare the curve.

Flask Calibration

The total flask volume is measured by weighing the amount of water required to fill the flask, measuring the water temperature, and dividing the water weight by the water density at the temperature used. Once the flasks are calibrated, they may be used for density measurements of scrubber liquors and slurries.

1. Label all flasks to be calibrated with permanent identification. Label the flask stopper with the same identification so that each flask will have its own unique stopper. Once labeled, never mix stoppers and flasks.

2. Clean flasks and stoppers and rinse well with deionized water. Oven dry flasks at 100°C and place in a desiccator to cool to room temperature.

3. Place each stopper in its respective flask, weigh to the nearest 0.01 g, and record the weight of the empty flask plus stopper on the data recording form (Figure F4-1, Section 13).

4. Carefully fill the flask completely with deionized water at laboratory temperature. Record the temperature of the water and its relative density as given in Table F4-2. Add enough deionized water so that the stopper may be placed in the flask with no air bubbles remaining below the stopper. Wipe the outside of the flask dry, weigh to the nearest 0.01 g, and record the weight.

Table F4-2

RELATIVE DENSITY OF WATER FROM 18°C TO 30°C

Temperature (°C)	Relative Density of Water
18	0.9986244
19	0.9984347
20	0.9982343
21	0.9980233
22	0.9978019
23	0.9975702
24	0.9973286
25	0.9970770
26	0.9968156
27	0.9965451
28	0.9962652
29	0.9959761
30	0.9956780

5. Repeat Steps 2 through 4 until three sets of weights are obtained for each flask being calibrated.

6. After the flasks are calibrated, clean, dry, and store the flasks and stoppers.

Slurry Solids Particle Density

Measure slurry solids particle density according to Section 12 of Method F3 in this handbook. Record the results as S on the data recording form (Figure F4-2, Section 13).

Slurry Liquor Density

1. Determine the density of filtered slurry liquor according to Method D1. Record density on the data recording form as L.

2. Record the flask volume on the data recording form for preparing the weight percent solids curve.

3. Follow the procedure outlined in Section 14 to prepare a curve relating weight percent solids to the density of slurry collected in the 100 mL flask.

Weight Percent Solids Determination

Determine the weight percent solids in a slurry sample as follows.

1. Select a sample flask for which the volume has been determined as described above. Record the flask ID, weight, and volume on the data recording form (Figure F4-3, Section 13). Completely fill the flask with slurry from the sampling port.

2. Insert the stopper without entrapping any air bubbles.

3. Wipe the flask dry and weigh to the nearest 0.01 g.

4. Record the weight of flask plus slurry on the data recording form.

5. Calculate the slurry density (see the data recording form in Figure F4-3, Section 13).

6. Use the curve prepared as described in Section 14 to determine weight percent solids in the slurry sample. Find the slurry density on the y-axis. Draw a horizontal line from this value to the curve. Read the weight percent solids on the x-axis directly below this point of intersection.

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For samples of the same slurry, follow Steps 1-6 (above) to obtain weight percent solids values from the same curve.

13.0   DATA RECORDING FORM

Refer to Figures F4-1, F4-2, and F4-3 for example data recording forms.

14.0   CALCULATIONS

Determine Slurry Density of Original Sample

$$C = \frac{Z}{F} \qquad (F4-1)$$

where: C = slurry density calculated from slurry weight and volume (g/mL),

Z = measured slurry weight (g), and

F = flask volume (mL).

The slurry density of the Hold Tank sample (Figure F4-3) is calculated as follows:

$$C = \frac{118.34}{112.70} = 1.050 \ g/mL$$

Data points needed to prepare a slurry density versus weight percent solids curve can be determined using the following calculation steps:

1.   Use the measured particle density of the solids (Method F3) to calculate the volume occupied by 5, 10, 15, 20, 25, and 30 g of solids:

$$V = \frac{W}{S} \qquad (F4-2)$$

where: V = volume of the slurry sample occupied by the solids (mL),

W = weight of solids (assume 5, 10, 15, 20, 25, or 30 g), and

S = particle density of slurry solids (g/mL).

Laboratory Name: _____   Analyst Initials: _____

Analysis Date: _____

Flask ID	R Weight of Flask plus Stopper plus Deionized Water (g)	E Weight of Empty Flask plus Stopper (g)	M[1] Weight of Deionized Water (g)	T Water Temperature (°C)	D[2] Water Density (g/mL)	F[3] Full Volume of Flask (mL)

[1] $M = (R - E)$

[2] D is from Table F4-2

[3] $F = \dfrac{M}{D}$

Figure F4-1. Flask Calibration Data Recording Form

Laboratory Name _____   Analyst Initials _____

Analysis Date. _____

S Measured particle density of slurry solids (g/cm³) = 2.607

L - Measured density of slurry liquor (g/ml)   = 0.9986

F - Volume of sample flask (ml)   = 113.77

Sample ID	W Weight of Solids (g)	V¹ Volume of Slurry Sample Occupied by Solids (mL)	R² Volume of Slurry Sample Occupied by liquor (mL)	Q³ Weight of Liquor in Slurry Sample (g)	Q + W Calculated Slurry Weight (g)	Y⁴ Slurry Density (g/ml)	X⁵ Weight Percent Solids (%)(W/W)
Calibration Slurry 1	5.0	1.918	111.85	111.70	116.70	1.026	4.28
Calibration Slurry 2	10	3.836	109.93	109.78	119.78	1.053	8.35
Calibration Slurry 3	15	5.754	108.02	107.86	122.86	1.080	12.21
Calibration Slurry 4	20	7.672	106.10	105.95	125.95	1.107	15.88
Calibration Slurry 5	25	9.590	104.18	104.03	129.03	1.134	19.38

$^1 V = W/S$

$^2 R = F - V$

$^3 Q = R \times L$

$^4 Y = \dfrac{Q + W}{F}$

$^5 X = [W/(Q + W)] \times 100$

Figure F4-2. Data Recording Form for Preparing Slurry Density Versus Weight Percent Solids Curve

F4-13

Laboratory Name: _____   Analyst Initials: _____

Analysis Date: _____

Sample ID	Flask ID	F Flask Volume (mL)	Weight of Flask plus Slurry (g)	Flask Weight (g)	Z Slurry Weight (g)	C[1] Slurry Density (g/mL)	Weight Percent Solids From Curve (%)
Hold Tank	2	112.70	178.96	60.62	118.34	1.050	8.0

[1] $C = Z/F$

Figure F4-3.   Data Recording Form for Weight Percent Solids by the Constant Volume Density Method

Record the resultant V for each W on the data recording form in Figure F4-2.

The volume occupied by the solids in Calibration Slurry 1 (Figure F4-2), assuming the weight of solids is 5.0 g, is:

$$V = \frac{5.0}{2.607} = 1.918 \text{ mL}$$

2. Calculate the volume of liquor in the flask by subtracting the volume of solids from the flask volume:

$$R = F - V \qquad\qquad (F4-3)$$

where: R = volume of liquor in slurry sample (mL), and
F and V are previously defined.

Record the resultant R for each W on the data recording form in Figure F4-2.

The volume occupied by the liquor in Calibration Slurry 1 (Figure F4-2), assuming the weight of solids is 5.0 g, is:

$$R = 113.77 - 1.918 = 111.85 \text{ mL}$$

3. Use the measured density of the slurry liquor (Method D1) to calculate th. eight of liquor:

$$Q = R \times L \qquad\qquad (F4-4)$$

where: Q = weight of liquor in slurry sample (g),
L = density of slurry liquor (g/mL), and
R = volume of liquor in slurry sample (mL) as previously def..ed.

Record the resultant Q for each W on the data recording form in Figure F4-2.

The weight of liquor in Calibration Slurry 1 (Figure F4-2), assuming the weight of solids is 5.0 g, is:

$$Q = 111.85 \times 0.9986 = 111.70 \text{ g}$$

4. Calculate the slurry density at each assumed weight of solids as follows:

$$Y = \frac{Q + W}{F} \qquad \text{(F4-5)}$$

where: Y = slurry density (g/mL), and
Q, W, and F are previously defined.

Record the resultant Y for each W on the data recording form in Figure F4-2.

The density of Calibration Slurry 1 (Figure F4-2), assuming the weight of solids is 5.0 g, is:

$$Y = \frac{111.70 + 5.0}{113.77} = 1.026 \text{ g/mL}$$

5. Calculate the weight percent solids at each assumed weight of solids as follows:

$$X = \left(\frac{W}{Q + W} \right) \times 100 \qquad \text{(F4-6)}$$

where: X = weight percent solids (%),
100 = factor to convert to percent basis, and
W and Q are previously defined.

Record the resultant X for each W on the data recording form in Figure 4-2.

The weight percent solids in Calibration Slurry 1 (Figure F4-2), assuming solids weight of 5.0 g, is found to be:

$$X = \frac{5.0}{111.70 + 5.0} \times 100 = 4.28\%$$

Now, prepare a plot (see Figure F4-4) of slurry density (y-axis) versus weight percent solids (x-axis). Use the curve to determine weight percent solids in the slurry sample. Find the slurry density, C (Eq. F4-1), of the sample on the y-axis. Draw a horizontal line from this value to the curve. Read the weight percent solids on the x-axis directly below the point of intersection. Do not use a linear regression fit.

15.0 PRECISION AND BIAS

The single-operator precision and bias of the constant volume density procedure given in Section 11 were determined in the laboratory. Slurries of ground limestone in saturated liquor at 50°C were prepared at 4.9 and 9.4 weight percent solids. Replicate determinations were performed on six samples of each slurry. Samples were obtained by pumping the slurry from a stirred, well-mixed vessel into the collection flask. The standard deviations of the weight percent solids for the 4.9 and 9.4 weight percent solids slurries were 0.012% and 0.016%, respectively. The bias for measured weight percent solids was 2.9% of the measured value for the slurry containing 4.9 weight percent solids and 4.0% of the measured value for the slurry containing 9.4 weight percent solids.

16.0 REFERENCES

1. 1977 Annual Book of ASTM Standards. Part 19. Standard Test Method for Specific Gravity of Soils, Method No. D 754. Philadelphia: American Society for Testing and Materials, 1977.

2. Determination of Density of Slurries and Liquors. Refer to Methods D1 and D2 of this handbook.

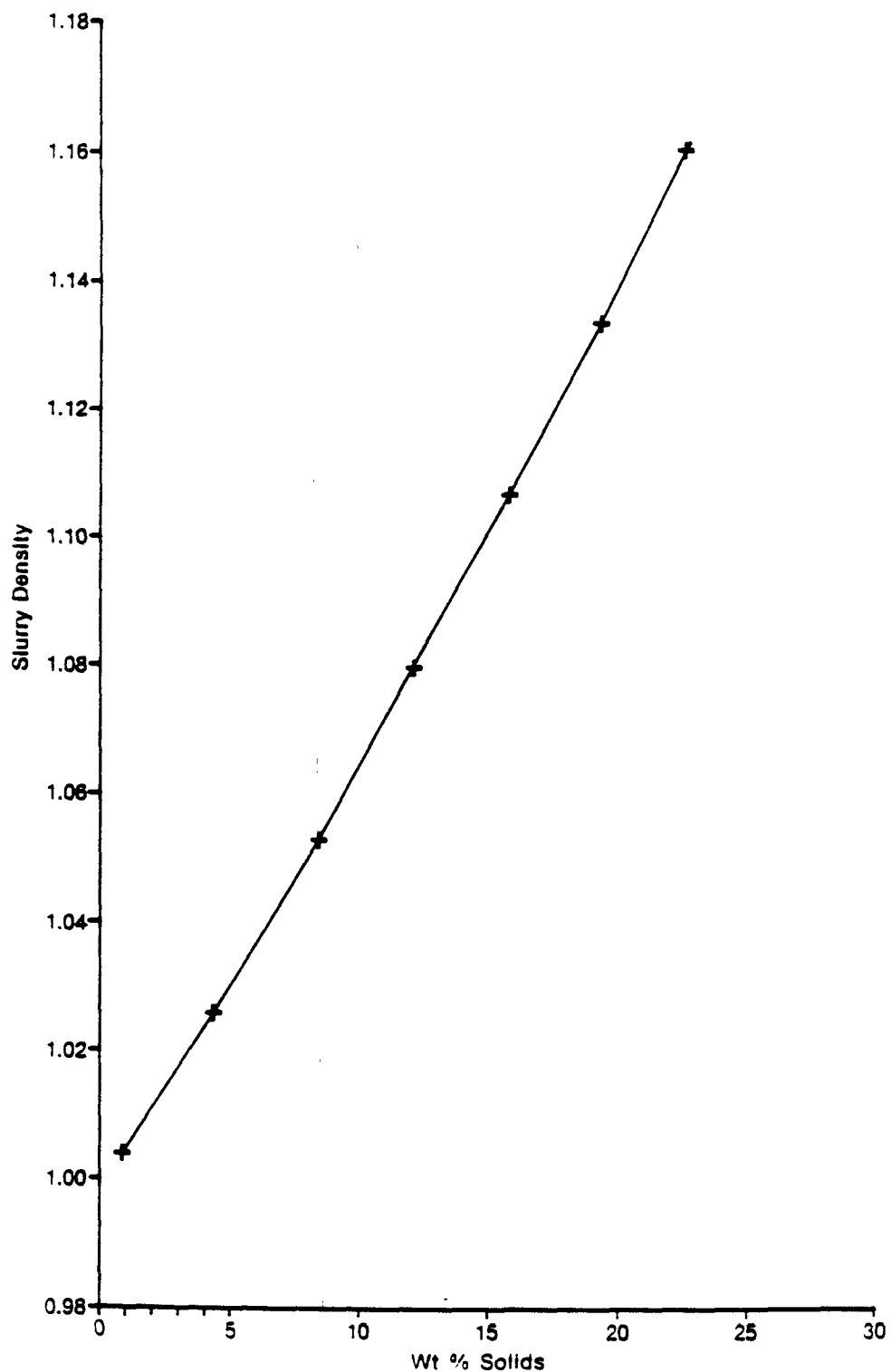3. A.E. Behl. Radian Laboratory Notebook No. 00739. March 1983.

Figure F4-4. Example Calibration Plot for Weight Percent Solids Determination by the Constant Volume Density Method